AMERICAN SOAPS, CANDLES, AND GLYCERIN.

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PREFACE.

THE writer, as a student in Boston, sought, but did not find, a technical treatise that dealt in a reliable and practical way with modern methods of soap manufacture and of the allied art of glycerin recovery. Numerous books on the subject of soap manufacture the writer at that time found interesting and to a degree instructive, but in later years, when engaged in practical work in the soap industry, the same books were found to be little less than worthless. The books were written by men of no practical knowledge of the art in which they presumed to instruct. Modern methods received no consideration. The information was second hand, ill digested, and misinterpreted, and for obsoleteness was equalled only by that of the mechanical appliances that were illustrated and described. A description of reasonable methods of glycerin recovery, to say nothing of reliability, considering the advance of the art, found no place. The demand, which always has existed and which has greatly increased with the growth of the industry, for a book that would be of practical value not to the practical man alone, but to the beginner, whether he be employer or employee, is the sole excuse for the present work.

The wide-awake practical man in any industry who is a thoughtful student of the trade literature of his art finds comparatively little help in the now voluminous literature which forms a class of its own called "technical." In a progressive art the technical treatises devoted to it are as a rule several years behind the most improved "shop" methods.

It is the experience of every one who has given the subject any consideration that the practical man who has something worth telling either from motives of his own will not, or from inherent deficiency cannot, write about it. The soap industry has been indebted for its technical literature to those who can write, but who have little if anything worth telling. The writer, admitting candidly that these are the conditions, submits this book to the consideration of the industry, realizing full well that while he may be classified as he himself has classified, he will receive that measure of approbation that his effort deserves.

This book had its inception when the writer was a student, and represents his fairly conscientious effort to supply the demand for such a book as he believes the trade desires. He has not attempted to teach the skilled and progressive soap maker anything that he does not now know. His art is one not learned from books. For such as he the writer has endeavored to supplement his practical knowledge by a systematized discussion of his art as presented in this book. It is to the beginner and to those in the industry who desire to supplement their practical knowledge that the writer's effort in the main has been directed.

The world is coming to realize as it has never done before that the highest privilege accorded to man is to help his fellow man. If this book should be instrumental in helping another, the writer will deem it an exercise of that high privilege, and therein will lie his reward.

This is a small world and all men are brothers. No man, however rare his accomplishments, knows it all. If by circumstance he be favored, he owes it as his duty as a man to share that favor, for thereby only can he keep it.

It is in this spirit that this book is submitted. It is the writer's contribution to the literature of an industry the status and progress of which in a most intimate sense indicate the degree of a nation's social development.

If this work, regardless of its defects, of which he alone is most conscious, may serve to advance the standard of practical usefulness of books on this subject, the writer's effort and influence will not be inconsiderable.

L. L. L.

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AMERICAN SOAPS, CANDLES, AND GLYCERIN.

CHAPTER I.

THE SOAP INDUSTRY.

Historical Considerations.

As American history is a graft upon the trunk, and a continuation in a new environment, of English traditions and customs, so is the industry whose technical study forms the subject of this work a continuation of the body of procedure which has its roots in English soil. But when we take into consideration the very primitive state of the industry during the formative period of the American nation, the industrial offshoot from the parent stem was too premature to be greatly influenced by parental characteristics. Save in a few isolated and, in their early days, unimportant instances, soap and candle-making was exclusively a household art, and continued so to be, despite the growth of systematized manufacture, until within the memory of many yet living. Soap manufacture as we know it to-day is a creation of the last fifty years. Setting aside the historically uncertain facts as to who discovered or first made soap, and confining our attention to our modern industrial environment, we need be concerned only with the fact that the extraction of lye from wood-ashes, Fig. 1, and its boiling up with the fat remnants of the kitchen were the duty of the early housewife; which simple procedure remained exclusively a household industry until urban growth, with its diversified interests, simple though they were in the beginning, had divorced certain portions of society from direct dependence upon the soil. At this time the house-to-house collection of fat-remnants and their rendering in an open kettle, the product of which was turned into soap and tallow candles, constituted the prosaic business of the early soap- and candle-maker. Candles were the first products of the tallow-chandler; soap acquired no prominence as an industrial product until Le Blanc, in 1791, under the exigencies of the French Revolution, devised a practicable method of making soda-ash from salt; previous to which time alkali was obtained almost entirely from wood-ashes and species of seaweed. The soap-manufacturer

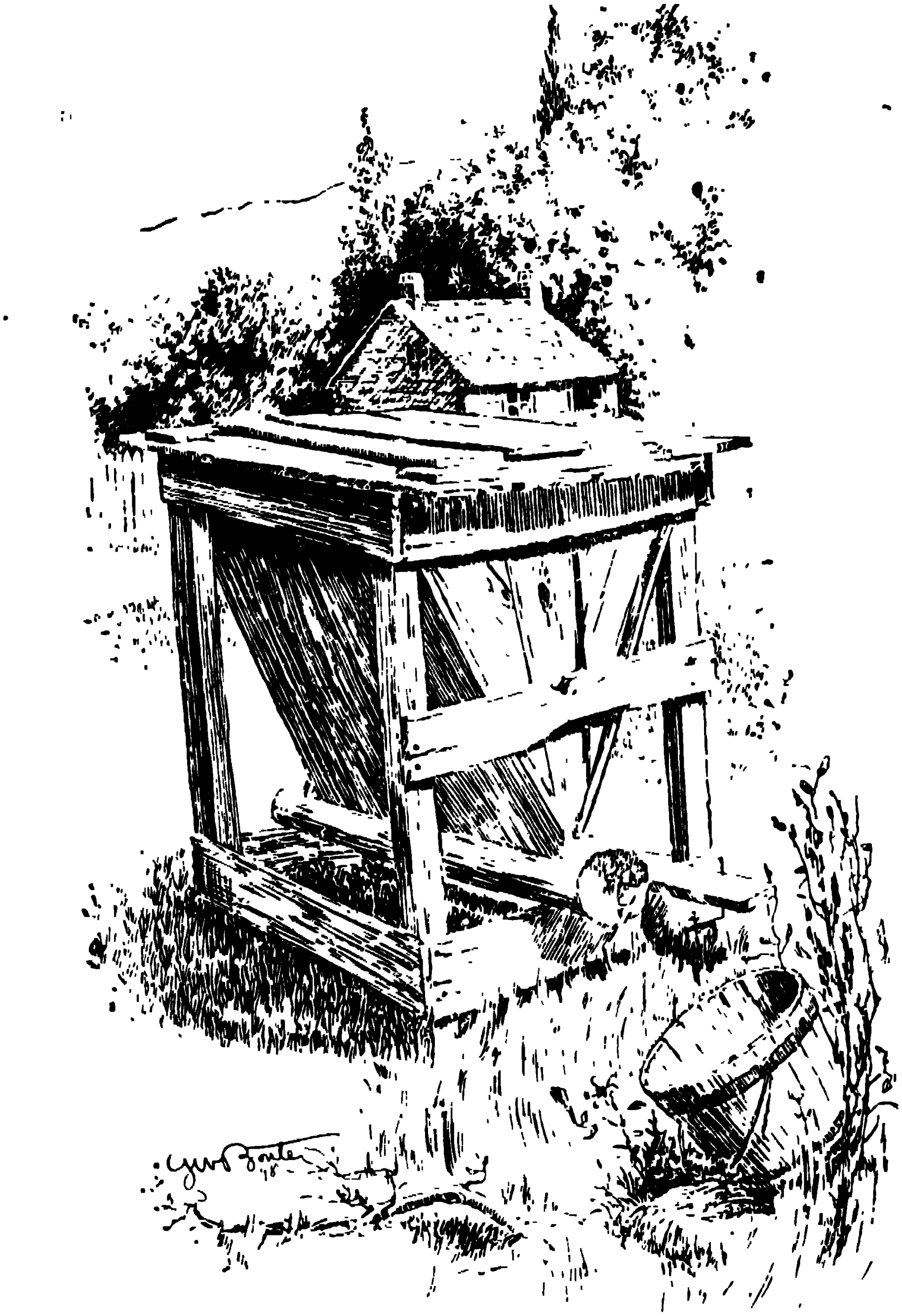

FIG. 1.—The Old-time Wood-ash Leaching Apparatus of the Farm.

was thus enabled to obtain his own caustic soda from soda-ash by causticizing with lime. This continued to be a part of the soap-manufacturing process for many years, and yet persists where, for reasons of cheapness, the volume of business transacted may warrant its employment. Artificial alkali was first used in the manufacture of soap in 1823. It was manufactured in England by James Muspratt according to the Le Blanc process. The Lancashire soap-boilers were loath to accept this new and purer article, and it was only after Muspratt had distributed gratis scores of tons of Le Blanc soda that they became convinced of the superior economy of artificial over natural soda. The commercial production of solid caustic dates from 1854, at which time improvements introduced in its manufacture by William Gossage in England resulted in its more general use, although in a very impure state, by soap-manufacturers and paper-makers. The use of the sheet-iron drums was introduced by Thompson in 1857, this innovation being a most welcome improvement over cooling the liquid caustic on iron slabs and subsequently breaking it into pieces and packing in barrels for shipment.

The Le Blanc process, a creation of French ingenuity, enjoyed its greatest development on English soil, and for half a century was one of the chief mainstays of England's industrial supremacy. The practical employment of the process involved certain technical and many unsanitary disadvantages, whose baneful effects proved a constant encouragement for the development of a simpler and more hygienic process. The mechanical difficulties surrounding the production of alkali by what is now known as the ammonia process met their first practical solution at the hands of Ernest and Alfred Solvay, and in the past thirty years there have been established, in every civilized country, works operating the process covered by the original Solvay patents. The first ammonia-soda works were established in Belgium in 1863. Later immense works were established in England by Brunner, Mond & Co., whose name is indissolubly associated with the development of the process in Great Britain.

In 1881 works for the production of alkali by the ammonia process were established by the Solvay Process Company near Syracuse. N. Y.

As a result of protection afforded by import duties, American-alkali manufacture increased by enormous strides, and not only completely divorced the United States from dependence upon English alkali, but lost this country as an export market for the coarser sodium salts of English manufacture.

Later similar works were established in the vicinity of Detroit, Mich., and at Akron, Ohio; and at Niagara Falls and elsewhere for the manufacture of caustic soda by electrolytic processes.

Tallow for many years was the sole soap stock; the use of cocoa-nut-oil in domestic-made soap dates from 1829; rosin as a soap-stock acquired prominence shortly before the Civil War; in 1862 the value of silicate of soda as an ingredient of household soap was first recognized in England; at about the same time A. Van Haagen of Philadelphia introduced the use of soda-ash in solution as a hardening agent for soft-bodied soap. During this period James Atkins of Brooklyn built the first soap-press, and about 1865 Babbitt put on the market the first cushion shaped pressed bar, and also in 1870 received one of the first patents for the recovery of glycerin from waste soap lye. Cottonseed-oil,* the first suggested outlet for which was as a soap stock, first attracted practical attention during this period, the general utilization of which, however, was delayed, owing to faulty processes of refining. A white, settled soap made buoyant by the incorporation of air acquired popularity as a "floating soap" early in the '80's.

In early days tallow was a farm product in the way that butter is to day. Farmers did their own butchering and preserving at certain seasons of the year, and it was only in localities adjacent to large centers that the animal carcass was sold and the separation of tallow from trimmings and scraps was made by the town butcher.

The purchase of cattle on hoof by local butchers and slaughtering was a general industry until the growth of large centers of population and the rapid absorption for agricultural purposes of land devoted to grazing.

The westward growth of population and the use of fertile prairie-land for cattle raising, with consequent wide separation from a ready market, proved an impetus to slaughtering near the source of supply. The modern Western packing house, following the center of corn production, is simply an economic substitute for the picturesque drover and the practice of transforming into edible products a rapidly deteriorating product at a remote distance from its source.

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* See "Cottonseed Products," by Lamborn.

That the packing and provision industry should come to be the largest producer of tallow is as natural a process as any organic development in biology which has efficiency for its unconscious aim.

The first soap kettles were of cast iron, of small capacity and fire-heated. The manufacture of sheet steel admitted of lighter construction and greater capacity. The contemporaneous introduction of steam as a heating agent gave the soap industry a great impetus in the direction of the use of mechanical appliances. The rotary pump displaced the bucket and dipper, and the mechanical mixer the hand crutch.

Frames of cast iron of greater capacity and portability displaced the sectional and detachable boxes of wood. Lighter and tighter frames of sheet steel marked an equal improvement over those of cast iron. The primitive method of slabbing by hand gave way to the time-saving slabber, which, though yet operated by hand, was a great improvement. The simple principle of the cutting-table restricted improvement to mechanical construction. The early foot press admitted of little essential improvement, which was soon supplied. As individual establishments grew in size, notably in Cincinnati, New York, Boston, Philadelphia, Baltimore, Detroit, Buffalo, Pittsburg, Zanesville, Chicago, and St. Louis, conditions requisite for the introduction of labor-saving machinery, viz., to assist in producing a larger output which can find a tolerably steady market, soon obtained. Boston, Buffalo and Brooklyn were the early centers of soap machinery manufacture and have retained their preeminence, although the manufacture of mills and plodders was early undertaken in Philadelphia, which was the birthplace of the milled-soap industry in the United States. Detroit, Pittsburg and Chester of recent years have assumed the prominence that has declined in Boston as a center for the manufacture of automatic soap-machinery, although in the latter city was the first complete line of automatic machinery offered to the soap industry. It included a slabber, a cutting table, and press, all of superior construction and recognized efficiency.

Milled soap acquired no prominence as a domestic product until after the Civil War, when the construction of the continuous helical plodder permitted a larger output than was possible with the hydraulic device previously in use. For the development of the milled-soap industry the reader is referred to the section devoted to that subject.

With the increasing prominence of trade-marks greater attention was directed to the appearance of the product. The first brand to acquire general prominence was the "German Detersive" of Jessie Oakley of Newburgh, N. Y. This was a cut pound bar, of a shape common in the early days but now little seen, and was wrapped by the manufacturer, or the wrappers were left loose in the top of each box. This form was a great advance over the method of supplying the grocer with large blocks of soap, from which it was cut as desired by the consumer. With the introduction by Babbitt of the cushion-shaped pressed bar the present characteristics of the soap industry were inaugurated. The stamping and wrapping of soap soon became general, in which connection is noteworthy the progress in die-construction and engraving. The multiplication of foot presses in large plants, with its incident disadvantages, encouraged mechanical ingenuity to develop the automatic press, which device has in very recent time been supplemented by the automatic soap-wrapping machine. In the manufacture of soap of low moisture content the practicability of substituting a continuous process for the laborious procedure, mechanical devices, and time consumed between the crutcher and the press, has been successfully demonstrated. The apparatus, the product of which is a milled soap of firm texture and high durability, has not as yet been applied to laundry soap-manufacture.

James Pyle was not only the first manufacturer of soap-powder in the United States, but as well the first general advertiser of note in the soap industry. Present-day soap advertising had its inception in Pyle's yielding to the persuasion of Horace Greeley to advertise in the *Tribune*. It is related that Greeley had sought for some time to secure an advertisement from Pyle. Finally he is reported to have said in substance: "Here is the rate-card. Use whatever space you wish for one year. If at the end of that time you find that it has paid to advertise, you may pay for whatever space you have used. If it hasn't paid, you need not pay." Subsequent events are eloquent of the confidence that each man had in the value of his respective commodity.

The last fifteen years are notable for the growth of soap-manufacture in the West and particularly as an allied interest of the packing and provision industry. Likewise in the South, a territory at one time the common market of Eastern manufacturers, the soap industry has grown apace with the establishment at large industrial centers of numerous prosperous concerns.

To close observers of conditions in the fat and oil industries, and to those in particular who can see the logical sequence of events from primitive conditions, when soap- and candle-making had barely emerged from the status of a household industry and when lard-making carried with it no import that it would ever rise above the industrial level of a by-product of the farm, it is apparent that a new era, more or less sharply differentiated from the old, is disclosed.

The line of demarcation, roughly but yet distinctly separating the old era from the new, is found in the order of their logical sequence and industrial relation, in the growth of pork- and beef-packing on a large scale, with the important features of artificial refrigeration and the utilization of waste products, the development of the alkali industry, the rapid growth of the cottonseed-oil industry, and the introduction among soap-manufacturers of the Jobbins and Van Ruymbeke process for the recovery of glycerin from waste soap-lye. Stimuli from without have not been lacking, chiefly among which are to be noted the advance in cheap and accessible transportation, on both land and sea, and what is most important, the social progress of the American people, which has expressed itself in an economic sense, in the vast increase of per-capita purchasing power. All industries in the United States have shared the same growth, and without doubt for each one can be noted an old era and a new one, differentiated chiefly by the application of the results of inventive ingenuity and the higher standard of executive intelligence following naturally as effect from cause. The growth of technical education as a business enterprise is a result of action and reaction, of supply and demand, operating between rapidly developing industries based upon scientific principles and of properly trained men demanded for their management. Confining our observations to the soap industry, it is apparent to every one that the first practical influence emanating from the source just stated arose from the general introduction of the Van Ruymbeke patents. While this factor not only brought a new class of men into the industry, it did what was more important: it raised the technical standard of executive men then in the industry. The point, however, to be noted in this connection is this, viz., that there is a different class of men in the soap industry to-day than there was thirty or as early as twenty years ago; and this change in the personnel of executive men is a direct reflection of the advanced social and industrial conditions of the period just noted.

Historically considered, the two great epochal events in soap-manufacture are the discovery of the Le Blanc process of soda-ash manufacture, whereby the industry was provided with the raw material of caustic soda of a purity and cheapness unknown before, and the discovery and application of a practical process for the recovery of glycerin.

For the former we are indebted to the inventive stimulus born of a great national necessity, aided by an alluring government prize during the early Napoleonic régime in France.

No invention is born in a night, or springs full-fledged, Minerva-like, from the head of Jove. The successful and practical form of an invention is the culmination of long continued effort on the part of many men. The Le Blanc process was originally so efficient that it has undergone no change from the form outlined by the inventor. Le Blanc wrested soda from refractory salt and gave it in its most active form to the industrial world.

For glycerin discovery we are indebted first to Chevreul, for it is due to him that soap-makers were first made aware of the valuable body present in a waste product of their art.

Glycerin, for many years, was made by decomposing fats, which process is now superseded by the process of recovering the glycerin and salt from the spent soap-lyes, the first United States letters patent for such a process being granted in 1870.

It is not our intention at this place to dwell upon the efforts that have been made commercially to recover glycerin from waste soap-lye. This history is written in patent specifications, each one introducing an idea or employing a mechanical principle that escaped a predecessor, and which brought the successive process a step nearer to commercial success, but the procedure of recovery was not greatly improved until the processes of Joseph Van Ruymbeke, as disclosed in United States Letters Patent issued to Jobbins & Van Ruymbeke.

These patented processes, now in general use throughout the world, represent the culmination—literally the survival of the fittest—in the strife of competing processes for the recovery of this valuable body. While this method of glycerin recovery in its outlines represents the maximum simplicity of procedure, it cannot be said that inventive ingenuity as regards apparatus never will supplant it.

The purification of waste lye, the concentration of the dilute purified liquor, with the ready separation of salt available for repeated use, and the distillation of the concentrated product, by means of apparatus of varying capacity and completeness according to the requirements of a given factory, constitute a process that is recognized as the best wherever soap is made and glycerin recovered.

It is unlikely that any general process ever will supplant the procedure of glycerin recovery that has been outlined, in connection with which the use of the apparatus referred to is an exclusive privilege.

The use of devices of different construction to effect the concentration of the purified waste lye and the distillation of the concentrated product in nowise is prohibited. Recent years have witnessed the successful application of such devices, notably those of Garrigues, whose ideas, likewise protected by patent, apply to the general process of glycerin recovery, embracing the purification of the waste lye, the concentration of the purified liquor, and the distillation of the concentrated product by a process and with apparatus devised by him.

The general problem of glycerin recovery, as it now confronts the soap manufacturer, displays an aspect which heretofore has been absent, viz., the privilege of selection on the part of the soap-manufacturer.

The last twenty years in the United States have witnessed a revolution in the management of the productive forces of industry. The development of the natural resources of a virgin continent resulted in a stupendous growth of the social power of consumption. Manufactures increased with unparalleled rapidity. It was inevitable that the resources of supply would surpass the possibilities of domestic consumption. The readjustment called for the highest acumen of modern business. The past era has been one of experimentation with the most potent forces of modern society which in their extent and potentiality were undreamed of before. These economic forces have been exercised along the line of natural law and have resulted in many lines of industry in the unification of executive control. The condition that has set these forces in operation is *competition*, and in no industry has it been keener than in the manufacture and sale of soap; but there is no industry on which these forces have left their impress so feebly.

In many industries producing commodities of general demand we find a large increase in the volume of output from a reduced number of productive units; evidence of the play of coöperative effort in manufacture with a consequent reduction of the evils incident to a régime of relentless competition.

In 1860 there were 530 establishments engaged in the manufacture of soap and candles; in 1870 there were 614; in 1880 there were 629; in 1890 there were 578; in 1900 there were 558. The diminution in the number of productive concerns arises from the dropping out of the ranks of those least fitted to survive and not from any combination of the persistent. The influences of the spirit of combination in the soap industry are found in the efforts of the prominent concerns to be self-sufficient and a unit unto themselves. We find this influence strongest in the packing and provision industry, where, it is true, soap is a secondary product in the slaughtering of cattle and the utilization of their by products. Among large soap manufacturers we find the absorption of allied industries as box-manufacture, printing, caustic soda manufacture, the production and refining of cocoanut- and cottonseed-oils, and the rendering of tallow. In the South soap-manufacture has been allied with the production of cottonseed-oil.

Instead of combination among soap-manufacturers we find, therefore, a marked tendency towards self-sufficient independence among the productive units and the localization of their markets. The result of such a tendency will be a reduction in the number of concerns by the more severe but none the less natural process of the elimination of the economically unfit.

But why has this process of progress been apparently decreed instead of the more economically humane method of combination? An answer must be sought in a consideration of the nature of the industry. As has been demonstrated, the most favorable conditions for the attainment of coöperative control of production in any industry consist in the control of the sources of the raw material of the industry and of the means of transportation; in fact these conditions to a high degree are essential.

The chief raw material of soap-manufacture is obtainable wherever meat is eaten. Substitutes are numerous and almost equally disseminated. Soap-making is a comparatively simple art, and necessary mechanical equipment for manufacture can be easily procured. These conditions are inconsistent with effective combination of productive effort. The market exists wherever the comfort and necessity of cleanliness are appreciated. A study of the application of the science of advertising to the selling of soap in connection with the subject of property rights in trade-marks should not be omitted in any general consideration of the soap industry. It is of great portent in the development of that kind of centralization which is peculiar to the industry. These simple, basic characteristics suffice to explain the economic conditions in the soap industry to-day.

CHAPTER II.

INTRODUCTION.

SOAP-MANUFACTURE is a chemical industry. In the various manufacturing processes compounds are used which represent broadly and strikingly the two grand divisions of the science of chemistry, viz., organic and inorganic chemistry. In fats and oils which receive, in the industry, the generic title of soap-stock are compounds which in their formation either in animal or plant are directly associated with vital activity. It is this characteristic that is fundamental with the bewildering multiplicity of compounds whose study constitutes organic chemistry. In the alkalis with which we are familiar, as caustic soda and soda-ash, are compounds of mineral origin, obtained primarily from common salt, which lie at the basis of our industrial civilization.

As soap-manufacture is so particularly a chemical industry, we cannot expect to attain to a thorough understanding of the art unless we familiarize ourselves, to some degree, not only with the formation of the bodies with which we work, but with their chemical peculiarities as well.

Chemistry teaches us that material nature is made up of simple elements which, in their combination in various proportions, constitute those bodies that are evident to our senses. Carbon, hydrogen, and oxygen are the three elements forming fats and oils, and if the soap-stock is pure they are the only elements that are present.

On the inorganic side we have the strongly basic element of sodium, which, in combination with oxygen and hydrogen to form the caustic alkali, or with carbon and oxygen to form the carbonated alkali, or with chlorine to form common salt, constitutes the essential inorganic raw material. In the following table is shown the chemical relation of the compounds which in their mixtures form fats and oils. There are also included certain quantitative characteristics which make chemistry an exact science. Soap chemistry on the organic side consists of the study of the chemical and physical properties of the compounds represented here.

There is no chemical distinction between a fat and an oil. The terms fat and oil as commonly used have reference to a physical state. By a fat is commonly understood a mixture of those glycerides that have a solid or semi-solid consistency at ordinary temperature and is usually of animal origin. An oil is usually liquid at ordinary temperature, although there are exceptions, and is either of animal or vegetable origin. Among the various fats and oils used in soap-manufacture, in a fat the glyceride stearin predominates; in an oil, the glyceride olein. In fats and oils of vegetable origin we find mixtures of the same glycerides that constitute animal fats. They possess the same chemical characteristics and are formed from the products of plant absorption under the influence of the same vital force. Paparelli, writing on the formation of the olive and its fatty substance, says:

"The pit is formed first, and increases rapidly in volume; and when it reaches its maximum weight the development of the flesh begins.

"The most important transformations which occur during the ripening of the olives are those that take place in the formation of the fatty substance in the flesh of the fruit.

"Although we do not yet know positively what substances are transformed into oil, nor the manner of this transformation, still we do know something of great utility in practical olive-culture, viz., the importance of the presence in the soil of alkali phosphates, potash, and lime for the formation of the oil in the olives; while nitrogen seems to favor especially the production of the wood and the formation of the pit.

"We know also that other conditions affect the formation and the quality of the oil, such as the location and the nature of the soil, climatic influences, olive varieties, the method of culture, the degree of maturity, and the composition of the fruit.

"Upon this subject I will only say that the quality and particularly the quantity of the oil depends especially upon the manner

TABLE I.—SATURATED FATTY ACIDS OF THE

Acid.	Formula.	Molecular Weight.	Per Cent NaOH Absorbed	Corresponding Glyceride.
Formic	$H \cdot COOH$	46	86.06	Formin
Acetic	$CH_3 \cdot COOH$	60	66.67	Acetin
Propionic	$C_2H_5 \cdot COOH$	74	54.06	Propionin
Butyric	$C_3H_7 \cdot COOH$	88	45.45	Butyrin
Valeric	$C_4H_9 \cdot COOH$	102	39.22	Valerin
Caproic	$C_5H_{11} \cdot COOH$	**116**	**34.48**	**Caproin**
Œnanthylic	$C_6H_{13} \cdot COOH$	130	30.77	Œnanthylin
Caprylic	$C_7H_{15} \cdot COOH$	**144**	**27.77**	**Caprylin**
Perlargonic	$C_8H_{17} \cdot COOH$	158	25.31	Perlargonin
Capric	$C_9H_{19} \cdot COOH$	**172**	**23.26**	**Caprin**
Undecylic	$C_{10}H_{21} \cdot COOH$	186	21.51	Undecylin
Lauric	$C_{11}H_{23} \cdot COOH$	**200**	**20.00**	**Laurin**
Tridecylic	$C_{12}H_{25} \cdot COOH$	214	18 27	Tridecylin
Myristic	$C_{13}H_{27} \cdot COOH$	**228**	**17 55**	**Myristin**
Pentadecatoic	$C_{14}H_{29} \cdot COOH$	242	16.53	Pentadecatoin
Palmitic	$C_{15}H_{31} \cdot COOH$	**256**	**15.63**	**Palmitin**
Daturic	$C_{16}H_{33} \cdot COOH$	270	14.81	Daturin
Stearic	$C_{17}H_{35} \cdot COOH$	**284**	**14 09**	**Stearin**
Nondecylic	$C_{18}H_{37} \cdot COOH$	298	13 43	Nondecylin
Arachidic	$C_{19}H_{39} \cdot COOH$	312	12.82	Arachidin
Medullic	$C_{20}H_{41} \cdot COOH$	326	12.28	Medullin
Behenic	$C_{21}H_{43} \cdot COOH$	340	11 77	Behenin
Carnaubic	$C_{23}H_{47} \cdot COOH$	368	10 87	. . . .
Hyænic	$C_{24}H_{49} \cdot COOH$	382	10 47	. . . .
Cerotic	$C_{26}H_{53} \cdot COOH$	410	9.76	. . . .
Melissic	$C_{29}H_{59} \cdot COOH$	452	8 85	. . . . . .
	UNSATURATED FATTY ACIDS OF THE OLEIC			
Acrylic	$C_2H_3 \cdot COOH$	72	55 55	. . . . .
Crotonic	$C_3H_5 \cdot COOH$	86	46 51	. . . .
(1) Angelic, (2) Tiglic	$C_4H_7 \cdot COOH$	100	40 00	. . . .
Hypogeic	$C_{15}H_{29} \cdot COOH$	254	15 75	. . . .
Oleic	$C_{17}H_{33} \cdot COOH$	**282**	**14 19**	**Olein**
	UNSATURATED FATTY ACIDS OF THE LINOLIC			
Elæomargaric	$C_{16}H_{29} \cdot COOH$	266	15 04	Elæomargarin
Linolic	$C_{17}H_{31} \cdot COOH$	280	14 20	Linolein
	UNSATURATED FATTY ACIDS OF THE LINOLENIC			
Linolenic	$C_{17}H_{29} \cdot COOH$	278	14.40	Linolenin
	UNSATURATED FATTY ACIDS OF THE RECINOLEIC			
Recinoleic	$C_{17}H_{32} \cdot OH \cdot COOH$	**298**	**13 43**	**Recinolein**

A. Acid occurs naturally in bodies of ants, in the stinging pine, and in certain nettles.
B. Occurs naturally only in slight quantities in the oil of *Enonymus Europæus.*
C. In cows' butter.
D. The acid occurs in animal and vegetable kingdoms both free and as an ester.
E. **In cows' butter, cocoanut- and palm-kernel oils.**
F. **In cows' butter, cocoanut- and palm-kernel oils.**
G. The acid occurs naturally in the leaves of *Perlargonium roseum.*
H. **In cows' butter, cocoanut- and palm-kernel oils.**
I. **In laurel, cocoanut- and palm-kernel oils and in spermaceti.**
J. **In Muscat butter, spermaceti, cocoanut- and palm-kernel oils.**
K. **Associated with stearin and olein in most animal and vegetable solid and liquid fats.**
L. Occurs in oil of seeds of thorn-apple, *Datura stramonium.*
M. **See Palmitin.**
N. Characteristic glyceride of peanut-oil.
O. Medullic acid (Thummel, Ber. '90) is a mixture of palmitic and stearic acids.

ACETIC SERIES. GENERAL FORMULA $C_nH_{2n+1}COOH$.

Formula.	Molecular Weight.	Per Cent NaOH Absorbed.	Per Cent Glycerol Set Free.	Per Cent Fatty Acids Set Free.	Natural Source. (See below.)
$C_3H_5(O\cdot HCO)_3$	176	68 10	52.28	78.41	A
$C_3H_5(O\cdot CH_3CO)_3$	218	55.04	42.20	82.56	B
$C_3H_5(O\cdot C_2H_5CO)_3$	260	46 15	35.38	86.14	C
$C_3H_5(O\cdot C_3H_7CO)_3$	302	39.74	30.47	87.42	C
$C_3H_5(O\cdot C_4H_9CO)_3$	344	34 88	26 74	88 94	D
$C_3H_5(O\cdot C_5H_{11}CO)_3$	**386**	**31 09**	**23.83**	**90 16**	E
$C_3H_5(O\cdot C_6H_{13}CO)_3$	428	28 04	21.50	91.14	
$C_3H_5(O\cdot C_7H_{15}CO)_3$	**470**	**25.54**	**19 57**	**91.91**	F
$C_3H_5(O\cdot C_8H_{17}CO)_3$	512	23 44	17 97	92.58	G
$C_3H_5(O\cdot C_9H_{19}CO)_3$	**554**	**21 66**	**16.61**	**93.13**	H
$C_3H_5(O\cdot C_{10}H_{21}CO)_3$	596	20 14	15 44	93 63	
$C_3H_5(O\cdot C_{11}H_{23}CO)_3$	**638**	**18 81**	**14 42**	**94 06**	I
$C_3H_5(O\cdot C_{12}H_{25}CO)_3$	680	17 65	13 53	94.41	
$C_3H_5(O\cdot C_{13}H_{27}CO)_3$	**722**	**16 63**	**12.75**	**94 75**	J
$C_3H_5(O\cdot C_{14}H_{29}CO)_3$	764	15 70	12 04	95 11	
$C_3H_5(O\cdot C_{15}H_{31}CO)_3$	**806**	**14.89**	**11 41**	**95.30**	K
$C_3H_5(O\cdot C_{16}H_{33}CO)_3$	848	14.16	10 85	95.52	L
$C_3H_5(O\cdot C_{17}H_{35}CO)_3$	**890**	**13 48**	**10 34**	**95 72**	M
$C_3H_5(O\cdot C_{18}H_{37}CO)_3$	932	12 87	9 87	95 92	
$C_3H_5(O\cdot C_{19}H_{39}CO)_3$	974	12.32	9.44	96 10	N
$C_3H_5(O\cdot C_{20}H_{41}CO)_3$	1016	11 81	9 06	96 27	O
$C_3H_5(O\cdot C_{21}H_{43}CO)_3$	1058	11.34	8 69	96 40	P
....	. .	.. . . .	..	....	Q
. ...	.. .	.	. . . .	......	R
.. ..	... .	....	. . .	.. ..	S
. .	.		....	....	T

SEPIES. GENERAL FORMULA $C_nH_{2n-1}COOH$.

.. ..	.	. .	. ...	..	U
. ...	.. .	.	.. ..		V
. . . .	.	.	....	..	W
$C_3H_5(O\cdot C_{15}H_{29}CO)_3$	800	15 00	11.49	95 26	X
$C_3H_5(O\cdot C_{17}H_{33}CO)_3$	**884**	**13 57**	**10 41**	**95.70**	Y

SERIES. GENERAL FORMULA $C_nH_{2n-3}COOH$.

$C_3H_5(O\cdot C_{16}H_{29}CO)_3$	638	14.35	11 01	95.45	Z
$C_3H_5(O\cdot C_{17}H_{31}CO)_3$	878	13 67	10.48	95.68	AA

SERIES. GENERAL FORMULA $C_nH_{2n-5}COOH$.

$C_3H_5(O\cdot C_{17}H_{29}CO)_3$	872	13.76	10.55	95.56	BB

SERIES. GENERAL FORMULA $C_nH_{2n-2}COOH$.

$C_3H_5(O\cdot C_{17}H_{32}\cdot OH\cdot CO)_3$	**932**	**12 87**	**9 87**	**95 92**	CC

P. Characteristic glyceride of oil of ben.
Q. Occurs as an ether in carnauba wax.
R. The acid occurs in the anal glandular pouches of the striped hyena.
S. The acid occurs in beeswax and in carnauba wax.
T. The acid occurs in beeswax.
U. Oxidation product of acrolein.
V. Occurs with isocratonic acid in crude pyroligneous acid.
W. Angelic acid is present in angelica-root. Tiglic acid occurs as a glyceride in croton-oil.
X. Present as a glyceride in peanut-oil.
Y. The characteristic constituent of liquid fats. See Palmitin.
Z. Occurs as a glyceride in Japanese wool-oil.
AA. Occurs as a glyceride in drying-oils.
BB. Occurs as a glyceride in drying-oils, notably linseed-oil.
CC. Characteristic glyceride of castor-oil.

in which the olive is cultivated—something requiring the greatest care."

Although the oil of the cottonseed and the peanut forms a structural part of the seed and does not constitute the fleshy portion of the envelope of the seed, as does olive-oil, the same remarks as to conditions affecting the formation of the oil are directly applicable.

Fats and oils are therefore indefinite mixtures of various glycerides whose chemical characteristics and the proportions in which they occur in the natural source determine the nature of the fat or oil. It will be evident that in order to understand the character of any fat or oil and its technical application, or in particular its applicability to soap-manufacture, we should be familiar with the chemical properties of its ingredients. Table I is composed of oxygenated compounds the formation of which can be traced from corresponding simple hydrocarbons. These series of hydrocarbons are termed homologous, i.e., there is a definite gradation in composition and in chemical and physical properties. This homologous character is retained in the oxygenated derivatives; for instance, formic acid is a very volatile liquid, boiling at 98° C.; acetic acid boils at 118° C. The complexity of the acid molecule increases as we ascend the series, and from fatty acids that are respectively liquid, oily, and of semi-solid consistency at ordinary temperature we reach those that are solid. The lower compounds of these homologous series are more active chemically than the higher ones and are more stable, as a result of their simpler constitution.

The higher compounds are of complex composition, of indifferent chemical activity, and are easily decomposed, breaking down under the action of heat into acids of simpler constitution, as in the development of rancidity, and of hydrocarbons, as in the formation of pitch in the distillation of fatty acids.

It will be noted that as the molecular weight of the fatty acids increases, the percentage of caustic soda required for saturation diminishes. This fact is likewise true of the corresponding glycerides shown in the adjoining column. A glyceride is a salt analogous to the salts of inorganic chemistry in that it is the result of the combination of an acid and a base. It is possible to form a glyceride in accordance with the following reaction:

Stearic acid + Glycerol Stearin + Water.

$$C_{17}H_{35}COOH + C_3H_5(OH)_3 = C_3H_5(O \cdot C_{17}H_{35}CO)_3 + 3H_2O.$$

The glyceride, acetin, is formed practically in the determination of glycerol in crude glycerin. Glycerides, however, are formed by nature by a process that the chemist is unable to trace. Corresponding with the decreased percentage absorption of caustic soda as the molecular weight of the glyceride increases, is the increased percentage of fatty acids set free on saponification with water, i.e., hydrolysis. Likewise with the increased yield of fatty acids by hydrolysis as the molecular weight increases, is the reduced percentage of glycerol. These tabulated facts have an important bearing in the manufacture of candle-stock and in the recovery of glycerin from waste lye in soap-manufacture.

The chief characteristic of normal soap-stock, with which the soap-maker is especially interested, is the percentage of alkali absorbed. The percentage absorption of alkali of various grades by the different stocks most commonly employed in American practice is shown in the following table:

TABLE II. POUNDS OF CAUSTIC SODA OF DIFFERENT GRADES REQUIRED TO SAPONIFY 100 POUNDS OF STOCK.

Soap-stock.	77½ Per Cent.	76 Per Cent.	74 Per Cent.
Tallow	13.7 –14.1	13.97–14.37	14.35–14.77
Cotton-oil	13.6 –14	13.87–14.28	14.24–14.67
Cocoanut-oil	17.5 –17.7	17.84–18.05	18.32–18.54
Olive-oil	13.6 –14	13.87–14.28	14.24–14.67
Palm-kernel oil	15.7 –17.7	16.01–18.05	16.44–18.04
Palm-oil	14 –14.4	14.28–14.60	14.67–15.09
Rosin	12.1 –14	12.34–14.28	12.68–14.67
	72 Per Cent.	70 Per Cent.	60 Per Cent.
Tallow	14.74–15.18	15.17–15.62	17.70–18.22
Cotton-oil	14.64–15.07	15.10–15.50	17.57–18.09
Cocoanut-oil	18.84–19.10	19.37–19.60	22.61–22.88
Olive-oil	14.64–15.07	15.10–15.50	17.57–18.09
Palm-kernel oil	16.90–19.10	17.39–19.60	16.12–22.88
Palm-oil	15.07–15.50	15.50–15.95	18.09–18.61
Rosin	13.03–15.07	13.40–15.50	16 –18.09

The variation in the amount of alkali absorbed by different fats and oils, likewise the variation in the amount of alkali absorbed by any particular fat or oil, arises from differences in the composition of the glycerides themselves, and from the varying proportions in which the glycerides occur in any particular sample. As we know that fats and oils are indefinite mixtures of various glycerides, the amount of alkali absorbed is influenced by the nature of the glycerides characteristic of the stock. The amount of alkali required for neutralization decreases as the molecular weight of the glyceride increases, hence we find that those commercial fats and oils in which glycerides of low molecular weight occur possess the highest alkali absorption. The increased quantity of salt required for graining such stock is due to the presence of those glycerides of low molecular weight, whose greater solubility in brine of the sodium salt is a marked characteristic. As the glycerides increase in molecular weight the solubility in brine of the soap obtained therefrom diminishes, hence less salt is required for graining. These analogies are shown in the following table, wherein is also indicated the yield of anhydrous soap from the glycerides mentioned:

TABLE III.—YIELD OF PRODUCTS FROM THE COMMERCIAL GLYCERIDES.

Glyceride and Chief Sources.	Yield on Saponification with Water.				Yield on Saponification with Caustic Soda.		
	Molecular Weight.	Per Cent Fatty Acids.	Per Cent Glycerol.	Per Cent Water Absorbed.	Per Cent Soap.	Per Cent Glycerol.	Per Cent NaOH Absorbed.
Laurin, cocoanut- and palm kernel oils. . .	638	94 04	14 42	8 46	104 38	14 42	18 80
Myristin, palm-kernel oil . .	722	94 47	12 70	7 17	103 87	12 70	16 57
Palmitin, palm-oil and tallow. . .	806	95 28	11 41	6 69	103 47	11 41	14 88
Linolein, linseed-oil .	878	95 68	10 48	6 16	103 19	10 48	13 67
Olein, olive- and cottonseed-oils.	884	95 70	10 40	6 10	103 16	10 40	13 56
Stearin, tallow and lard. . . .	890	95 73	10 34	6 07	103 14	10 34	13 48
Recinolein, castor-oil	932	95 92	9 88	5 80	103 00	9 88	12 88
Arachidin, peanut-oil.	974	96 10	9 44	5.44	102.87	9 44	12 31

In Table IV, compiled from various sources, are given the percentages of caustic potash and caustic soda required to saponify the various fats and oils mentioned:

TABLE IV.—ALKALI ABSORPTION OF FATS AND OILS.

Fat or Oil.	Per Cent KOH Absorbed.	Per Cent NaOH Absorbed	Fat or Oil.	Per Cent KOH Absorbed.	Per Cent NaOH Absorbed.
Lard-oil	19.1-19.6	13.6-14	Peanut-oil	19.1-19.6	13.6-14
Almond-oil	19.5-19.6	13.9-14	Cottonseed-oil	19.1-19.6	13.6-14
Sesame-oil	19 -19.4	13.5-13.7	Linseed-oil	18.7-19.5	13.3-14
Rape-oil	17.1-17.9	12.2-12.8	Hempseed-oil	19.3	13.8
Poppyseed-oil	19.3-19.5	13.8-14	Walnut-oil	19.6	14
Niger-seed oil	18.9-19.1	13.5-13.6	Seal-oil	18.9-19.6	13.5-14
Whale-oil	18.8-22.4	13.4-16	Porpoise-oil	21.6	15.5
Butter	22.1-23.2	15.8-16.5	Cocoanut-oil	24.6-26.8	17.5-17.7
Palm kernel oil	22 -26.7	15.7-1[illegible].7	Lard-oil	19.2-19.6	13.7-14
Tallow	19.3-19.8	1[illegible].[illegible]-14.1	Bone-fat and grease	19.1-19.7	13.6-14
Sperm-oil	12.3-14	8.8-10	Castor-oil	17.6-18.1	12.5-13
Palm-oil	19.6-20.2	14 -14.4	Cod-oil	18.5-21.3	13.2-15.1
Olive-oil	19.1-19.6	13.6-14	Rosin	17 -19.3	12.1-14

We have seen that a glyceride is a representative of that class of bodies called salts. These bodies are neutral in their character, being neither acid nor alkaline. Stearin, for instance, is glyceryl stearate, $C_3H_5(O \cdot C_{17}H_{35}CO)_3$. Glycerol does not exist as such in neutral glycerides, but is formed by the absorption of the elements of water in the process of saponification.

The term "saponification" applies to a vast number of chemical changes whereby a fatty acid, or similar body, is set free and an alcoholiform body produced. The saponifying agent may be water, in which case the acid body set free is a pure fatty acid; if an alkali, the fatty acid is in combination with the saponifying agent and constitutes soap. When the saponifying agent is water, the reaction is commonly called "hydrolysis."

Accurate knowledge of the chemistry of saponification dates only from 1813, at which time Chevreul determined the chemical constitution of fats and oils and the nature of the effects produced upon them by alkalis. Chevreul demonstrated that ordinary fats and oils are chemical compounds consisting of a base, glycerol, and of different acids, termed generically "fatty acids," but specifically stearic, palmitic, and oleic, forming respectively, in combination with glycerol, the bodies, stearin, palmitin, and olein. In aqueous saponification, stearin, which may be taken as representative of tallow, yields 95.73 per cent of fatty acids and 10.34 per cent of glycerol, through the absorption of 6.07 per cent of water. These

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numerical relations will be plainly evident from the following equations and proportions:

Stearin (890). Water (54). Glycerol (92). Stearic acid (852).

$$C_3H_5(O \cdot C_{18}H_{35}O)_3 + 3H_2O = C_3H_5(OH)_3 + 3C_{18}H_{36}O_2.$$

Stearin (890) : glycerol (92) : : 100 : x;

$x = 10.337 +$ per cent. glycerol yielded.

Stearin (890) : stearic acid (852) : : 100 : x;

$x = 95.72$ per cent. fatty acids yielded.

This simple arithmetic lies at the foundation of the autoclave or digester processes for the manufacture of fatty acids for candle-stock. In saponification with alkali, the latter remains in combination with the fatty acids and constitutes anhydrous soap. The percentage-absorption of alkali and yields of glycerol and anhydrous soap are represented as follows:

Stearin (890). Caustic soda (120). Glycerol (92). Anh. sodium stearate (918).

$$C_3H_5(O \cdot C_{18}H_{35}O)_3 + 3NaOH = C_3H_5(OH)_3 + 3C_{18}H_{35}O_2Na.$$

Stearin (890) : anh. Na stearate (918) : : 100 : x;

$x = 103.15$ parts anh. Na stearate from 100 parts of stearin.

Stearin (890) : caustic soda (120) : : 100 : x;

$x = 13.49$ per cent caustic soda required for saponification.

Stearin (890) : : glycerol (92) : : 100 : x;

$x = 10.337$ per cent glycerol yielded.

This simple arithmetic lies at the basis of the art of soap-boiling.

By reference to Table I it will be seen that by adding together the figures representing respectively the percentages of fatty acids set free and glycerol formed, and subtracting from this sum 100, the percentage of water absorbed in the hydrolysis of any glyceride mentioned may be ascertained.

Likewise in Table III, by subtracting 100 from the amounts of anhydrous soap and glycerol formed from 100 parts of the glyceride mentioned, the percentage of sodium hydrate absorbed in saponification may be learned.

Soap, then, is simply a mixture of the alkali salts of the fatty acids, and the process of effecting combination is, in the language of the kettle-room, soap-boiling.

The term "soap" admits of a variety of applications. We are concerned solely with that alkali compound of the fatty acids which is soluble in water; but there is a number of other compounds of the fatty acids with heavy metals which are insoluble in water and which receive the name soap. Aluminum soap is used as a thickener of lubricating-oils. Conrady (*Apoth. Zeit.*, 14, 767) recommends aluminum oleate prepared by precipitating dissolved aluminum sulphate with common-soap solution, with the addition of 10.30 per cent of petroleum, as a product of the consistency of vaseline suitable for impregnating leather. Iron and chromium soaps are used in dyeing and color-printing. The "driers" of varnish-manufacture are the insoluble compounds of lead and manganese with linseed-oil. They are also used in the manufacture of oilcloth, in waterproofing canvas, and in the preparation of leather substitutes. Their chief characteristic is their insolubility in water, which effectually bars them from use as a detergent.

According to the purpose for which the soap is intended, the numerous varieties found on the American market may be divided into three general classes, viz.:

Domestic soap—including all varieties of detergents having soap as a base and used for domestic or general cleansing purposes;

Toilet-soap—including all variety of soap used for the toilet and bath;

Industrial soap— including all varieties of detergents having soap as a base and used in the laundry and textile industries.

Soap as a cleansing agent is essentially a vehicle for the slow and progressive liberation of the caustic alkali which is present in a combined state in the neutral soap. In the discussion of the properties of the fatty acids represented in Table I, we learned that the lower members of the series are volatile and soluble in water and that the solubility diminishes as the molecular weight increases, until we reach complete insolubility with stearic acid. These same properties hold for the corresponding soaps or alkali salts of the fatty acids. There has been no contribution of equal value to our knowledge of the chemistry of fatty bodies since Chevreul's investigations during the early part of the last century. The results of his researches, which were published in 1823,* still remain the most important source of our knowledge of the cleansing action of soap.

Until Chevreul's researches (1811-1823) it was believed that soap consisted simply of a binary compound of fat and alkali. Claude J. Geoffray in 1741 pointed out that the "fat" or "oil" recovered from soap solutions by neutralization with mineral acid differs from the original fatty substance by dissolving readily in alcohol, which is not the case with ordinary fats and oils. The significance of this observation was overlooked, and equally unheeded was a no less important discovery by Scheele in 1783, that fats and oils contain a sweet principle now known as glycerin. The discoveries of Geoffray and Scheele formed the basis of Chevreul's researches, by which he laid bare the constitution of fats and oils and the true nature of soap.

The effective action of soap is based on its employment in a state of aqueous solution. As Krafft and Stern have demonstrated—in harmony with the views of Chevreul—soap, when brought into contact with water, under suitable conditions of concentration and temperature, is decomposed into free fatty acids and free alkali, in addition to which a portion of still unaltered soap passes into solution. On cooling, the free fatty acids unite with the dissolved soap, hot solutions producing a somewhat insoluble acid fatty-acid salt, while conversely, if cold water had been used in dissolving, the latter is left behind from the outset. These facts are the outcome of experiments conducted by Krafft with chemically pure alkali salts of the high molecular fatty acids present, in the form of glycerides, in fats and oils. The decomposition of soap into basic and acid soaps on contact with water, as reported by Rotondi, Fricke, and others, does not actually occur; and the views held on this matter by those workers are explained by Krafft, by the simple circumstance that neutral alkali oleate, unlike the corresponding palmitates and stearates, are but slightly decomposable by water. Hence, in case of the commercial soaps, which generally contain all three classes of compounds, the cold-water extract will be found to contain neutral oleates and free alkali—forming the hypothetical basic fatty-acid salt—while the free fatty acids constitute, with the neutral salts of the solid fatty acids, the insoluble constituents of the soap. Thus, in washing with soap, four factors come under consideration: first, the free alkali; secondly, the free fatty acid; thirdly, the dissolved neutral fatty-acid salts; and lastly, the acid fatty-acid compound formed by contact with the free fatty acid.

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*Recherches chimiques sur les corps gras d'origine animale. (Republished in 1889.)

If it is permissible to regard the operation of washing as a process of emulsifying the fats which everywhere are the cause of adherent dirt, and which are thus forced into combination and thereby removed, this object can be satisfactorily attained by means of the four factors above referred to, in conjunction with water and mechanical motion—rubbing, beating, rinsing. In the first place the high moistening capacity of the soap solution—which power is probably attributable to traces of eliminated alkali—facilitates penetration into the material to be washed, driving out the air and gaining access to the greasy dirt. The task of removing the latter is shared by the undecomposed, neutral, fatty acid salts which emulsify the fat, and by the acid fatty acid salts—the lather—which then envelop the no longer coherent particles of dirt, keep them in suspension, and enable them to be got rid of by simple rinsing. The alkali set free, which, in case of the neutral soaps, is very small in quantity under the usual conditions of applying the soap in a solid state (soaping the clothes), plays a very inferior part in the washing process, far below that formerly ascribed to it in connection with the action of soap. In fact, it has little influence, even in dilute solutions of soap, since the liberation of alkali and degree of dilution are almost proportional; and, moreover, because the state of emulsion prevailing during the washing process ameliorates the effect otherwise exerted by free alkali on animal and vegetable fibres. These fatty emulsions exert an anti-friction influence in conjunction with the melted free fatty acids present in hot solutions of soap; and the beautifying effect obtained by washing with soap is further contributed to by the considerably reduced mechanical effort necessary to effect the removal of dirt when the lubricating action of soap comes into play.

Hillyer writing recently on the cleansing power of soap * reviews the various theories to account for the cleansing action of soap solutions that have been presented since Chevreul's time, and advances the theory that the emulsifying power of the soap solution is due to the soap itself and not to the alkali liberated by hydrolysis. He states:

Chevreul, the first to study the fats and soaps, discovered that when soaps are acted on by water they are hydrolyzed to free alkali and to an acid salt. The tendency of the reaction may be expressed by the equation

$$NaAc + H_2O = NaOH + HAc,$$

where Ac stands for the palmitic or stearic acid radical. The acid thus set free unites with more or less of the undecomposed soap to form difficultly soluble acid salts which contain more and more acid in proportion to the alkali as the dilution of the solutions from which the salts separate increases.

Rotondi, basing his conclusions on a mixed soap, decided that when water acts on the soap, there is formed an acid salt which is soluble with difficulty in cold water and a basic salt which is easily soluble. Recent works by Krafft and his coworkers † have shown that Chevreul was correct and that Rotondi fell into error, probably because he did not take into account the fact that sodium oleate is readily soluble in cold water. It was probably a mixture of this with the alkali set free by the hydrolysis of the palmitate and stearate present which was called an alkaline soap or basic salt. The more current theories of the detergent action of soap are largely based on the undoubted hydrolysis of soaps by much cold water.

The most ordinary theory is that the alkali set free by the hydrolysis of the soap acts on the fat to remove it by a process of saponification. To show that this is illogical it is only necessary to call attention to the fact that the alkali present has the opportunity of making a soap in either one of two ways: First, by acting on the

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* Jour. Am. Chem Soc, May, 1903.

† Ber. d. chem. Ges., 27, 1747; ibid., 27, 1755.

glyceride to decompose it, or, secondly, by acting on the fatty acid or acid salt from which it has just been separated. That it will more readily react with the latter than with the former is clear, and since, while the dilution is great, this reaction with the acid salt does not take place but rather the reverse reaction, it seems entirely improbable that the hydrolytic alkali acts chemically, if at all, on the glycerides of the fats and oils. In the use of soap for cleansing, we prefer to use hot and strong solutions, but the hydrolysis is almost certainly more complete when the solutions are dilute and have cooled enough to yield a precipitate of acid salt. In the hot solution, especially if concentrated, there can be but little alkali free. Finally, paraffin-oils are washed away by soaps as well as the glycerides are, and in this case it is clear that there can be no chemical reaction of the alkali upon the oil.

C. R. Alder Wright * assumes that the value of a soap is largely due to the alkali of hydrolysis acting in such a way as to allow contact of the water with the substance to be cleansed. Ladenburg's "Handwörterbuch," Vol. X, p. 574, favors the theory of Knapp, which lays the cleansing power of soap to the property of the soap itself of easily wetting oily substances. Knapp's original papers are not available to the writer † and his evidence is not known, but it may be easily shown by a simple experiment that it is the soap itself and not the alkali which gives aqueous solutions the power of wetting oily substances.

A piece of red litmus paper is thoroughly oiled with either purified cottonseed oil or a paraffin-oil, and upon it is placed a drop of dilute alkali. The alkali produces a blue spot with well-defined edges and spreads very slowly over the oiled paper. If a drop of soap solution is placed on the paper it spreads rapidly and soon produces a large spot with ill-defined ragged outlines.

Another suggestion as to the cleansing is that the soap itself or the alkali of hydrolysis acts as a lubricant, making the tissue and impurities less adhesive to one another, and in that way promoting the removal of the latter. It is not unreasonable to consider this as a factor in the cleansing, and it will be referred to again later.

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* Muir's Dictionary of Applied Chemistry, Vol. III, p. 411.

† Hillyer, Jour. Am. Chem. Soc., May, 1903.

It is often suggested, either as a distinct theory or as a more or less important adjunct to the saponification theory, that the alkali set free by hydrolysis acts on the fat by emulsifying it and carrying it away in suspension with the other impurities. This suggestion has come apparently because those who have emulsified oils with alkali have used oils not free from fatty acids. It is also suggested that the emulsification is due to the undecomposed soap, but experiments are not recorded, as far as can be ascertained, to sustain this view or to determine whether the action is due to the soap or to the alkali.

When good cottonseed-oil, "salad oil," is shaken with weak alkali it is largely emulsified, but by washing the emulsion with water and dilute alkali alternately for many times, the soap formed by the action of the alkali on the free acid of the oil is removed, the oil separates from the emulsion, and a product is obtained which is not emulsified by decinormal sodium hydroxide. Neither is kerosene emulsified by decinormal alkali. Both kerosene and purified cottonseed-oil easily make permanent emulsions with a decinormal solution of sodium oleate. From this experiment it seems certain that the emulsifying power of the soap solution is due to the soap itself rather than to the hydrolytic alkali.

The explanation of the action of soap as due to its emulsifying power is a plausible one, but no clear account has been given, or made current, at least, in chemical literature, of the physical properties which a liquid must have to be a good emulsifying agent. The great similarity between foams and emulsions in method of making and in properties suggests the question whether any explanation given for the formation and permanence of foams will not also apply to the formation and permanence of emulsions. Plateau and Quincke have made extensive studies of substances which foam and of those which are emulsifying agents. There will be here no attempt to detail their results or to follow exactly their reasoning, but the general trend of their work will be indicated. They have shown that soap solution has a surface-tension which is lower than that of any other aqueous solution. Its surface-tension is, in fact, only about 40 per cent that of pure water. This may be shown in several ways. When two capillary tubes of the same diameter are placed respectively in water and in soap solution, the water will be seen to rise about 2.6 times as high as the soap solution. If a given volume of water is allowed to drop from a broad-ended pipette or stalagmometer and then a soap solution is allowed to drop from the same pipette, it will be found that the soap solution will make 2.6 times as many drops as the water. The cohesion of the soap solution is so small that the surface-tension will sustain drops of a volume only about 40 per cent of the volume of those formed by water. Plateau * lays the power of forming bubbles, films, and foam to two factors: first, the liquid must possess notable viscosity, that the film may not readily yield to the forces which tend to thin it to the point of rupture; secondly, it must have a low surface-tension, since the surface-tension is the most active force in thinning the film. Soap solutions are ordinarily quite viscous, and this viscosity is increased in Plateau's bubble mixture by adding glycerin, which aids in making bubbles and films permanent. Soap solutions have a low surface-tension, and on account of these two factors soap solutions easily yield and enduringly maintain films and foam.

Quincke † has similar fundamental views, but ascribes the permanence of a foam to the mixed character of the liquid which foams, and claims that no pure liquid will foam. According to Quincke the permanence of the foam is due to the action of surface tension which spreads out over the surface of the film some secondary ingredient of the solution, and this tends to close up any potential ruptures in the film. Plateau calls attention to the important part played by viscosity in these phenomena and speaks of the existence of a viscosity which pertains to the liquid itself, its internal viscosity, caused by the friction of the molecules of the liquid on each other, and also of a second kind of viscosity, which he calls superficial viscosity, which sometimes makes the motion of a foreign body upon the surface of a liquid more or, on the other hand, less ready than within the liquid. Stables and Wilson ‡ have confirmed Plateau's work, and find that the motion of a body in the surface of a solu-

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* Pogg. Ann., 141, 44.

† Wiedemann's Ann., 35, 502.

‡ Phil. Mag. (5), 15, 406.

tion of saponin is resisted 600 times as much as it is within the same solution. By this great superficial viscosity they account for the great foaming power of saponin solution, although it has a comparatively high surface-tension.

To make these ideas clear a simple case may be used as an illustration. Suppose two bubbles of air to be lying side by side within a mass of water. The molecules of water at the point of nearest approach of the bubbles are acted on by the stress of the surface-tension of both bubbles, and this causes a thinning of the film between them. The surface-tension is great and the water is mobile, so that the thinning will be rapid and soon the bubbles will coalesce by the breaking down of the membrane. If, instead of water, soap solution is present, the stress which causes thinning will be less on account of the much smaller surface-tension and the resistance to thinning, due to the viscosity of the soap solution, will be somewhat greater and as a result the bubbles will remain separate for a much longer time. In the same way a mass of bubbles will remain permanent longer in soap solution than in water.

Can emulsification be explained by the viscosity of the emulsifying agent? In the case of very thick liquids like the gum solutions used by the pharmacists, it is probable that the extreme viscosity is a very large factor in giving permanence to the separation of the oil-droplets which have been formed mechanically. That even very large internal viscosity will not make an emulsion permanent is shown by the fact that 50 per cent glycerin and 6 per cent gum solutions which have viscosity of a high degree will not emulsify kerosene or even a viscous oil like cottonseed-oil. Saponin or albumen solution with their high superficial viscosity will give permanent emulsions. Dilute soap solutions which have not great viscosity have very great emulsifying power, and the same is true to a less degree of some other solutions. From these facts it must be decided that internal viscosity of the liquid will not account for its emulsifying power.

It has been seen that foaming can be explained largely by the small surface-tension of soap solution towards air. Surface-tension phenomena show themselves between two liquids as well as between a liquid and air. The question arises whether there is any peculiarity in the degree of surface-tension between soap solutions and oils not shown by other solutions.

Quincke* observes that when a solution of sodium carbonate is brought in contact with an ordinary oil, phenomena are exhibited which he ascribes to the change of surface-tension between the oil and aqueous solution on account of the formation of soap. Among the phenomena accounted for in this way is that of emulsification.

From his numerous experiments demonstrating his views Hillyer concludes that the low surface-tension and the emulsifying power are due not to the alkali or to the acid salt set free by the hydrolysis, but to the *undecomposed soap itself.*

We have seen before that another possible factor in cleansing, namely, the power which soap solutions have of wetting oily substances, is due to the soap itself. But this wetting power may be explained also by the strong adhesion of the soap to the oil and the low cohesion of the soap solution itself. The latter will more easily be spread out over the surface if its cohesion is small, and a stronger force will be acting to spread it out if it is strongly attracted to the oily surface.

A very similar factor in cleansing is that which may be called ease of penetration, by which the aqueous solution pushes into and permeates the interstices of the fabric. If a glass tube of 3 or 4 mm. internal diameter and closed at one end is filled with cottonseed-oil and immersed in a vessel of water, the oil will not leave the tube, being held there by the strong surface-tension film between the oil and water. But if a strong solution of soap is poured in, the surface-tension film is diminished in strength, the oil flows from the tube, and the soap solution penetrates into it. Here the soap, on account of its weak adhesion and its strong adhesion to the oil, withdraws the oil and penetrates into the oily tube. In the same way, it may be, the soap penetrates into the capillary interstices of the fabric to be cleansed.

Even the lubricating power of soap solutions may be explained by considering the factors a low cohesion and a strong adhesion. In treatises on mechanical engineering, the chief physical property mentioned as desirable in a lubricant is that it shall have sufficient "body" or viscosity to prevent its being pressed out from between the surfaces to be lubricated. While viscosity is for this particular object no doubt a necessary thing, it would seem to be a necessary evil. As the term is used in physics, viscosity is a property of fluids which prevents the freedom of motion which their particles would have if the fluid had no viscosity or a less viscosity. But in speaking of lubricants mechanical engineers demand, in a substance intended to promote motion, a property which physically hinders motion. If we consider as lubricants graphite or steatite or soap, or the clay on which our shoes slip in the street, we may get a clearer notion of lubrication. In these cases at least the lubrication is due to the fact that the lubricant adheres in a film to the surfaces to be lubricated but does not cohere to itself. In the use of oil as a lubricant, it is also true that the oil adheres to the lubricated surfaces and soon forms two layers of oil which glide over one another. We have then strong adhesion on the part of lubricant and a weak cohesion. The same is true of soap solutions and will explain their slipperiness and lubricating power. The slipperiness we feel when alkali is used on the hands is probably due to its action on the skin or on the material which keeps in place the epithelial cells. The actual removal of these cells with the impurities attached to them and covered by them would account for the cleansing action of alkali and alkali carbonate on the skin.

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* Wiedemann's Ann., 35, 580.

The position here taken is then: that the cleansing power of soap is largely or entirely to be explained by the power which it has of emulsifying oily substances; of wetting and penetrating into oily textures; and of lubricating texture and impurities so that they may be removed easily. It is thought that all of these properties may be explained by taking into account the low cohesion of the soap solutions and their strong attraction, adhesion, or affinity to oily matter, which together cause the low surface-tension between soap solution and oil.

Hillyer in this study of soap solutions * reports the agreement of his conclusions with well-known facts in regard to soaps as follows:

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* Jour. Am. Chem. Soc., May, 1903.

"Sodium oleate is very soluble in cold water and even in dilute solutions shows little hydrolysis. Soaps rich in oleate are useful for toilet purposes and for wool-scouring when cold water is used. Its stability in dilute solutions would make it of value as a detergent even until it is completely rinsed away, and its ready solubility would make it easy to wash away. For laundry work, dish-washing, and other work, when hot water is used practical experience shows that the most desirable soap is a tallow soap. This is rich in palmitate and stearate and some oleate. The palmitate and stearate have a very high efficiency at a high temperature, especially when the concentration is great. When the concentration is lessened marked hydrolysis takes place and the efficiency rapidly falls off, but with mixed soaps this low efficiency of the palmitate and stearate is supplemented by the relatively high efficiency of the oleate in dilute solutions, which will sustain the detergent effect until all impurities are washed away, including the acid palmitate and stearate, which might otherwise be retained by the fabric. When the temperature is low, palmitate and stearate are so little soluble as to be of no practical value, since the only effect of water on them is to hydrolyze them and set free a small quantity of alkali, which, according to the hypothesis here favored, has no detergent effect on neutral oils. Rosin soap is usually regarded as a comparatively undesirable ingredient in soaps. By the test employed by Hillyer it is of about the same efficiency as sodium oleate, but in dilute solutions, especially in the heat, it shows marked hydrolysis, which is necessarily accompanied by the separation of the rosin acids. Here the acid product of hydrolysis separates in a cloud and does not stay in solution, as do the palmitate and stearate, in the heat. The separated rosin acids may well settle on the fabric being washed and impart to it the odor of rosin, cause it to be yellow and make it ready to easily take up the dust. This effect may be partially offset, in using mixed soaps, by the other ingredients, whose detergent action will tend to remove the rosin acids, but the evil effect will still to some extent remain."

The fitness of a soap for cleansing purposes is determined therefore by the nature of the material to which the soap is applied, the nature of the dirt to be removed, and the conditions under which the process of cleansing is carried out. No soap satisfies a number of requirements with equal success. Each general purpose demands a soap of a certain character that adapts it for the most satisfactory work.

CHAPTER III.

RAW MATERIALS OF SOAP-MANUFACTURE.

Classification of Raw Materials. Animal Fats and Oils. Beef-fat. Oleo-stearin. Oleo-oil. Tallow. Hog-fat. Lard-stearin and Lard-oil. Classification of Animal Soap-stock. Red Oil. Handling of Tallow in the Factory. Soap-stock of Vegetable Origin. Olive-oil. Olive-oil Foots. Linseed-oil. Cottonseed-oil. Cottonseed-oil Soap-stock Corn-oil. Cocoanut-oil. Palm-oil. Palm-kernel Oil. Peanut-oil. Castor-oil. Rosin. Vegetable Soap-stock of Minor Importance. The Alkali Industry. Caustic Soda. Commercial Grading of Soda-ash and Caustic Soda. Causticization of Soda-ash by the Soap-manufacturer. Causticization of Soda-ash. Description of Plant. Procedure of Causticization. Manufacture of Sal-soda. Potassium Carbonate and Caustic Potash. Salt. Filling Materials. Silicate of Soda. Borax. Talc. Mineral Soap-stock. Starch.

Classification of Raw Materials.—In the following diagram are represented the chief raw materials of soap-manufacture. They are classified with respect to their origin and to their chemical or mechanical relation to the finished product. This diagram serves as a brief introduction to an extended examination of the origin, production, characteristics, and use in the soap industry of the various products thus classified

- Chief raw materials of soap-manufacture
  - Organic.
    - Animal....
      - Beef-fat.
      - Hog-fat.
    - Vegetable....
      - Cottonseed-oil.
      - Rosin.
      - Cocoanut-oil.
  - Inorganic.
    - Caustic soda—in chemical combination with soap-stock.
    - Soda-ash........ } in mechanical admixture with soap.
    - Silicate of soda... } in mechanical admixture with soap.

FIG 2.—Classification of Raw Materials.

Animal Fats.—Fat exists in nature in the form of minute globules disseminated among the tissues of the animals and seeds of the vegetables producing it. To separate it in a commercial form heat and pressure are required.

The fat-cells, always present in a nearly spherical form (as shown at *a* in Fig. 3), at intervals are traversed by a minute network of blood-vessels from which they derive their secretion—*b* are fibres of areolar tissue.

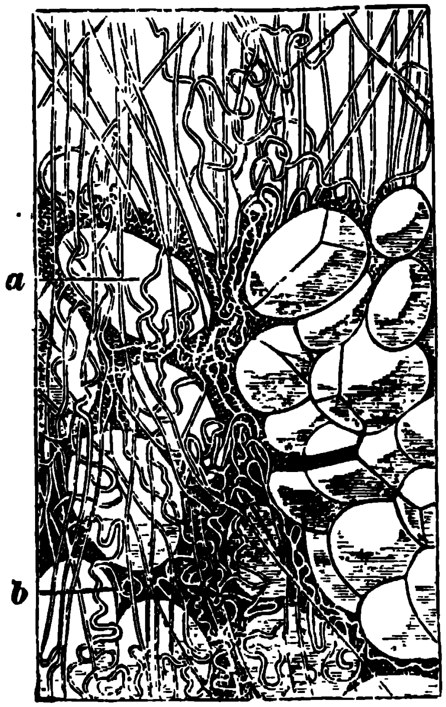

FIG. 3.—Fatty Tissue of Animals (Magnified).

Fatty tissue of animals, of which the microscopic appearance is given in the cut, cannot be broken up by pressure; but when heated so as to expel all its water, the animal tissue contracts and shrivels up at the same time the fat-globules are much expanded, so that the cells burst and the fat flows out in a liquid mass.

The fat of animals is derived partly without change by the absorption of the fat of foods and partly from proteids and carbohydrates forming the essential ingredients of the food. While the direct absorption of fat from foods by the organism forms no inconsiderable source of the natural body, proteids and carbohydrates may be considered the raw materials of natural fats made in and stored up by the organism. When the food contains an excess of fat or of those ingredients essential to its formation, the amount above what is necessary to serve as fuel for the vital machine is stored up in the fatty tissue, which reserve becomes available and is quickly exhausted when the animal is confined to food deficient in fats and fat-forming materials.

Fat occurs in all organs and tissues of the animal in extremely variable amounts. The largest proportional amount is contained in bone-marrow, this substance containing over 960 parts of fat in 1000. The chief deposits of fat in the animal are the intermuscular connective tissue, fatty tissue of the abdominal cavity, and the subcutaneous connective tissue. The fatty tissue, being of low conducting power, retards loss of heat by radiation; it serves as a reserve food-supply and to protect and support certain internal organs. The less water present in the fatty tissue, the richer it is in fat. Schultze and Reinecke found in one thousand parts of fatty tissue of the origin stated the following proportions of water, membrane, and fat:

	Water.	Membrane.	Fat.
Fatty tissue of oxen.........	99.6	11.6	888.8
" " " sheep.........	104.8	16.4	878.8
" " " swine.........	64.4	13.5	922.1

Fat gradually varies in composition from the exterior to the interior parts of the same animal. The fat immediately below the skin has the lowest melting point and that in the middle of the body the highest, while the melting-point of the fat in the intermediate parts varies with its distance from the interior. Not only does the composition of fat from different tissues of the same animal vary within wide limits, but also that from the same tissues in different species of animals. The fat present in the fat-cells consists chiefly of the glycerides of stearic, palmitic, and oleic acids, and in fats present as animal secretions, as butter-fat, the glycerides of caproic, capric, and caprylic acids occur.

Beef-fat.—Before edible uses for beef fat had arisen, as in the manufacture of lard compound and oleomargine, all fat rendered from the tissues of cattle was known commercially as tallow. This increased consumption for edible purposes has placed the demand for tallow for soap manufacture in a secondary place and compelled the acceptance of inferior grades of tallow for the latter purpose.

The caul fat of beef yields on rendering in open kettles at low temperature oleo-stearin and oleo oil. This fat is known as "butter" stock. The kidney fat as a rule remains with the carcass and is known as suet. Trimmings of the carcass and portions of the viscera form the raw material of tallow. Its suitability for lard compound determines its classification. If not good enough for lard compound, its outlet is the soap-kettle. Tallow varies greatly

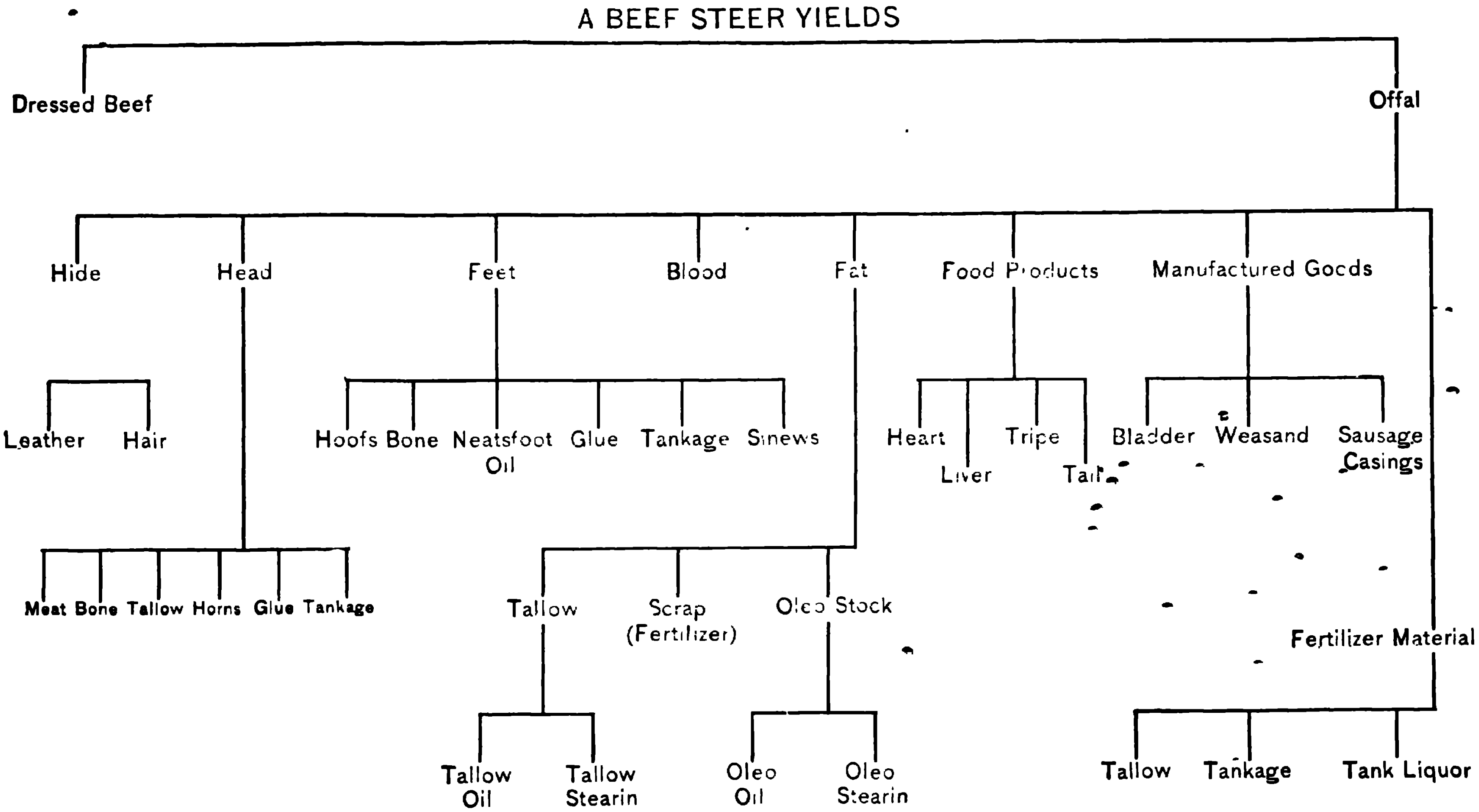

FIG. 4.—Diagram of Beef Products.

in flavor according to the quality of the stock and the conditions of rendering. It varies in color from white to brown under the same general conditions. Tallow from fresh and carefully rendered stock should not have a greater fatty-acid content than 1.5 per cent, although

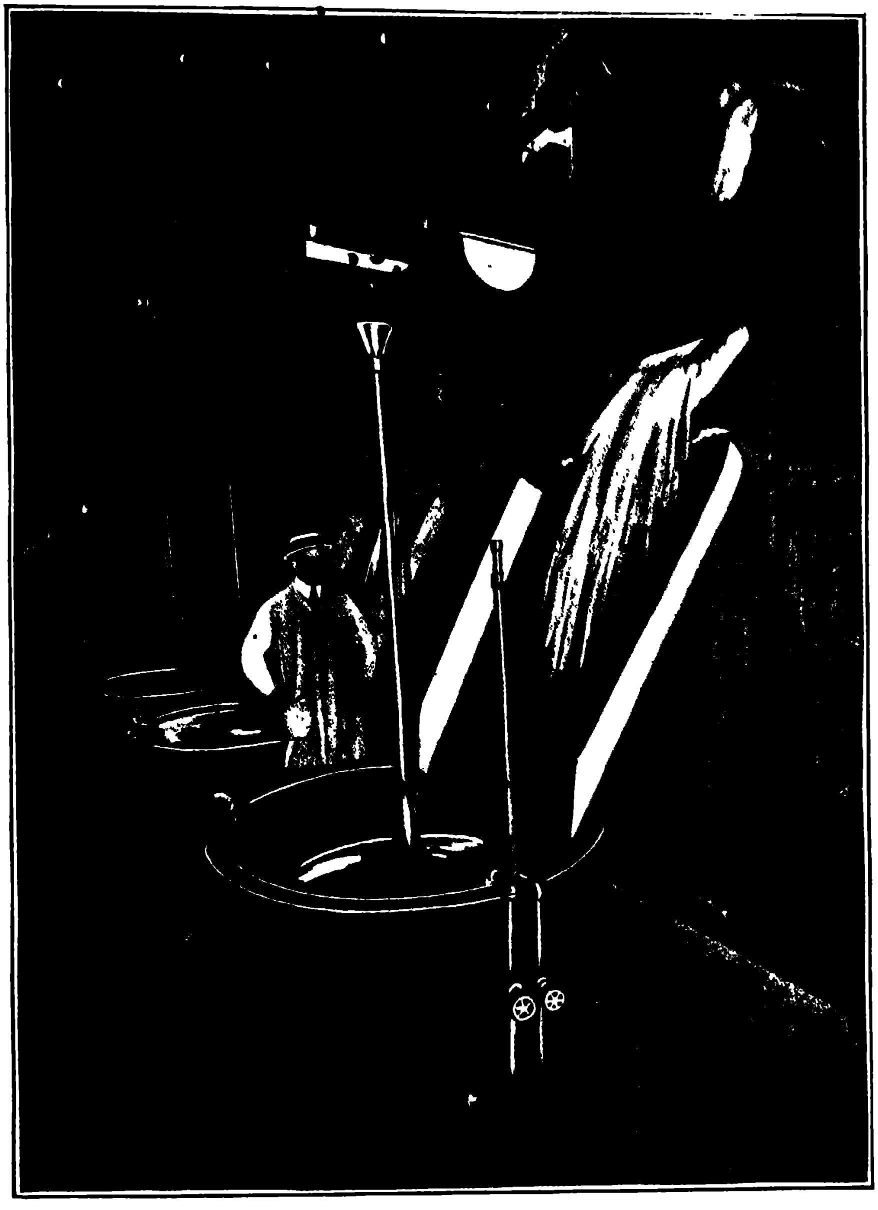

FIG. 5.—Melting Beef-fat for Oleo-oil and Oleo-stearin.

as a rule it runs from 2 to 10 per cent. Three per cent of free fatty acids is the maximum for edible tallow. An acidity approaching 5 per cent or more unfits it for edible purposes. As all grades of beef-fat are used in soap manufacture according to the quality and purpose of the soap, brief descriptions of their processes of manufacture will be given.

Oleo-stearin and Oleo-oil.—After the animal is slaughtered the fat is removed and placed in a vat of warm water, where it is thoroughly washed to remove blood and adhering impurities. It is then chilled and hardened in a bath of ice-water, after which it is finely comminuted by cutting-machines and melted in steam-jacketed caldrons at a temperature of about 160° Fahr. Slowly revolving agitators keep the fat moving until the melting process is complete, when the whole is allowed to settle. Fig. 5 represents a car of comminuted fat from the cutting-machine being dumped into the melting kettle. The settling process is accelerated by the addition of salt, which is scattered over the entire surface of the liquid and settles the fibre, or "scrap," to the bottom. After the first settling the clear oil is carefully siphoned to a second series of jacketed caldrons, usually on the floor below (see Fig. 6), where more salt is added, and the temperature controlled until a second settling is completed. This demembranized fat is now siphoned into mounted vats and allowed to stand from three to five days at a temperature favorable to the crystallization of the stearin, a part of which forms a crust over the top, and the remainder settles to the bottom, leaving the clear oil between. It is a common phenomenon in the crystalli-

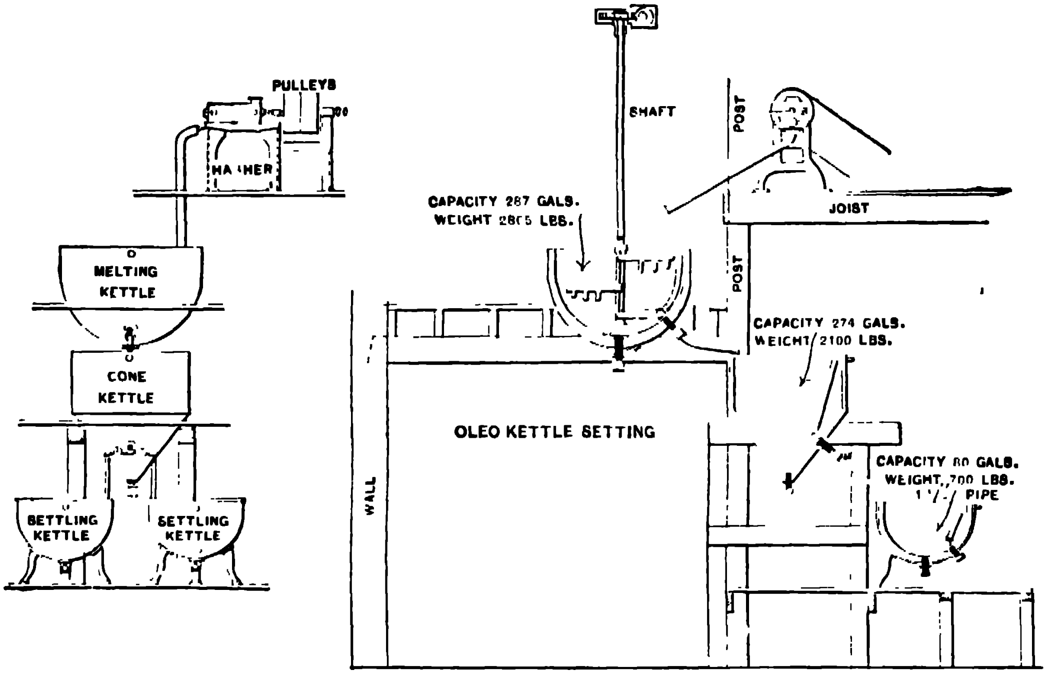

FIG. 6.—Diagram of Oleo-plant.

zation of various substances whose specific gravity is not greatly in excess of the mother-liquor, that, cooling first at the top, a portion of the substance which is being crystallized out forms a crust over the surface and the remaining portion is precipitated. When the vats have stood the required time the crust is broken into fine particles and the whole is given a thorough mechanical mixing which leaves it of a mushy consistency. It is then wheeled to a revolving table surrounded by skilled workmen, who wrap the mixture into small packages of canvas cloths—each containing about 3 pounds—which are built into the presses. The oleo-oil is then separated by great pressure, slowly and gradually applied, and flows from the presses into a large receiving-tank on the floor below (see Fig. 7). The stearin remains as a compact cake and is used with cottonseed-oil in the manufacture of lard compound. The oleo-oil goes into oleomargarine. Caul fat treated as described yields about 50 per cent oleo-oil, about 30 per cent stearin, and the remainder shrink-

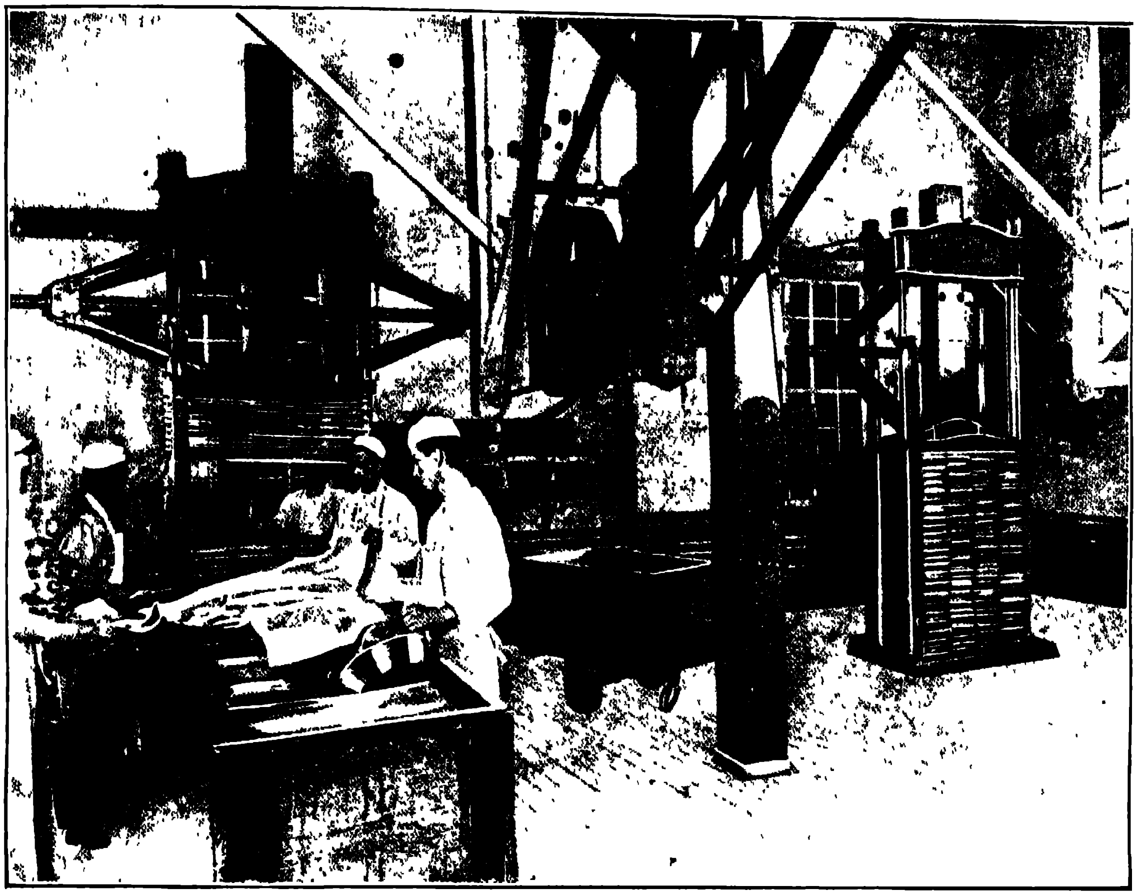

FIG. 7. -Separating Oleo-oil from the Stearin of the Fat by Pressure.

age. These yields vary greatly according to the amount of tissue and the temperature of pressing.

Tallow.—Those parts of the fat-secreting portions of beef that are converted into soap-stock are made up of that quality which unfits them for edible purposes. With the development of the provision and allied interests, the utilization of animal fats for food has increased to the degree that only the poorest quality is available for soap manufacture. The fat is contained in the fat-cells, which with membrane and water make up the fatty tissue. The process of separating fat from the enclosing membrane is called "rendering," and is generally effected by steam under pressure in closed tanks. Rendering in open kettles at low temperatures has been described in the manufacture of "butter" stock. Steam-rendering is employed for all stock, whatever its origin, that will not yield comestible products. The type of the modern rendering-tank as used in the packing industry is shown in Figs. 8 and 9. These tanks are made of tightly riveted steel plates capable of withstanding an average minimum steam pressure of 50 pounds. They are usually 5 to 6 feet in diameter and 10 to 16 feet in height with a corresponding capacity of 1500 to 3000 gallons. The largest ones are 8 feet in diameter and 20 feet high. They are equipped with safety-valve and suitable pipe connections for filling and emptying. Modern tanks discharge through a gate-valve at the bottom which is of sufficient size to give an unobstructed opening. They are made of ⅜-inch steel and doubly riveted on the sides and are carefully calked inside and out to prevent leakage from corrosion. The cone-shaped ends, carrying, respectively, the charging manhole and the drop-valve, or door, as the case may be, are of ⅛ inch steel. In addition to the charging manhole on the top are two air-cocks to carry away offensive fumes formed during "cooking," and a safety-valve. On the sides are three draw-off cocks for the removal by gravity of the rendered fat. Steam is introduced through a pipe entering the bottom cone.

In charging the tank with material to be rendered it is filled within a foot of the junction of the upper cone and cylindrical portion of the tank. The charging door is tightly closed and a single vent-pipe opened. Steam at 40 pounds pressure is now admitted.

The length of time required to satisfactorily "cook" the stock depends upon the amount and character of the charge. A tank with a yielding capacity of 25 tierces is generally 6 feet in diameter and 12 feet high and requires from eight to ten hours for cooking. No water is added during the rendering period, sufficient resulting from the condensation of the steam. Should this increase excessively, which condition can be ascertained by the rendered stock blowing

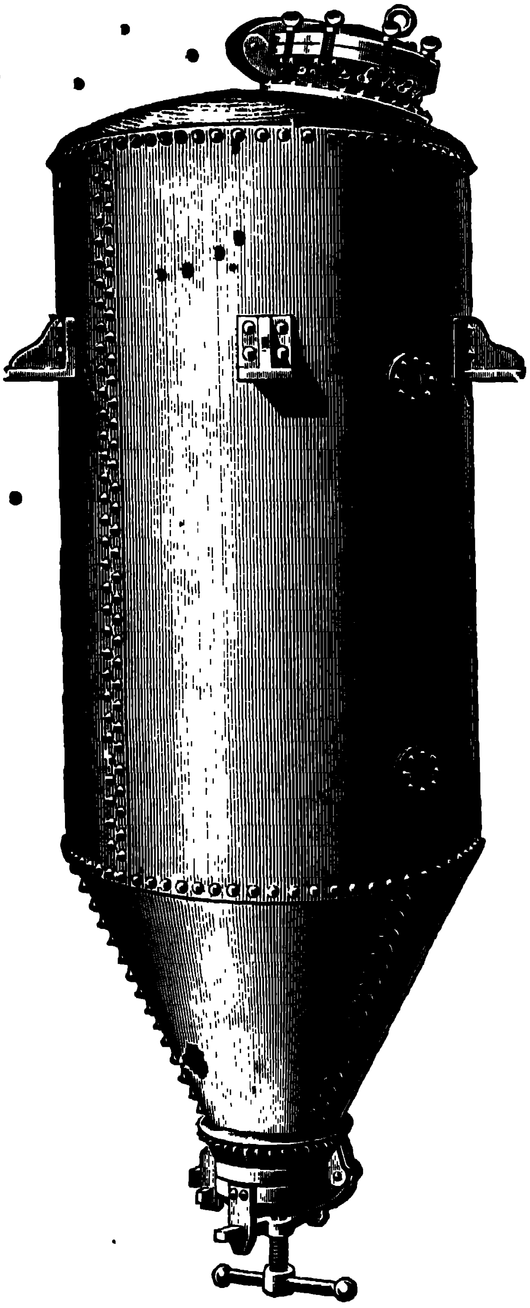

FIG. 8.—Packers' Rendering-tank.

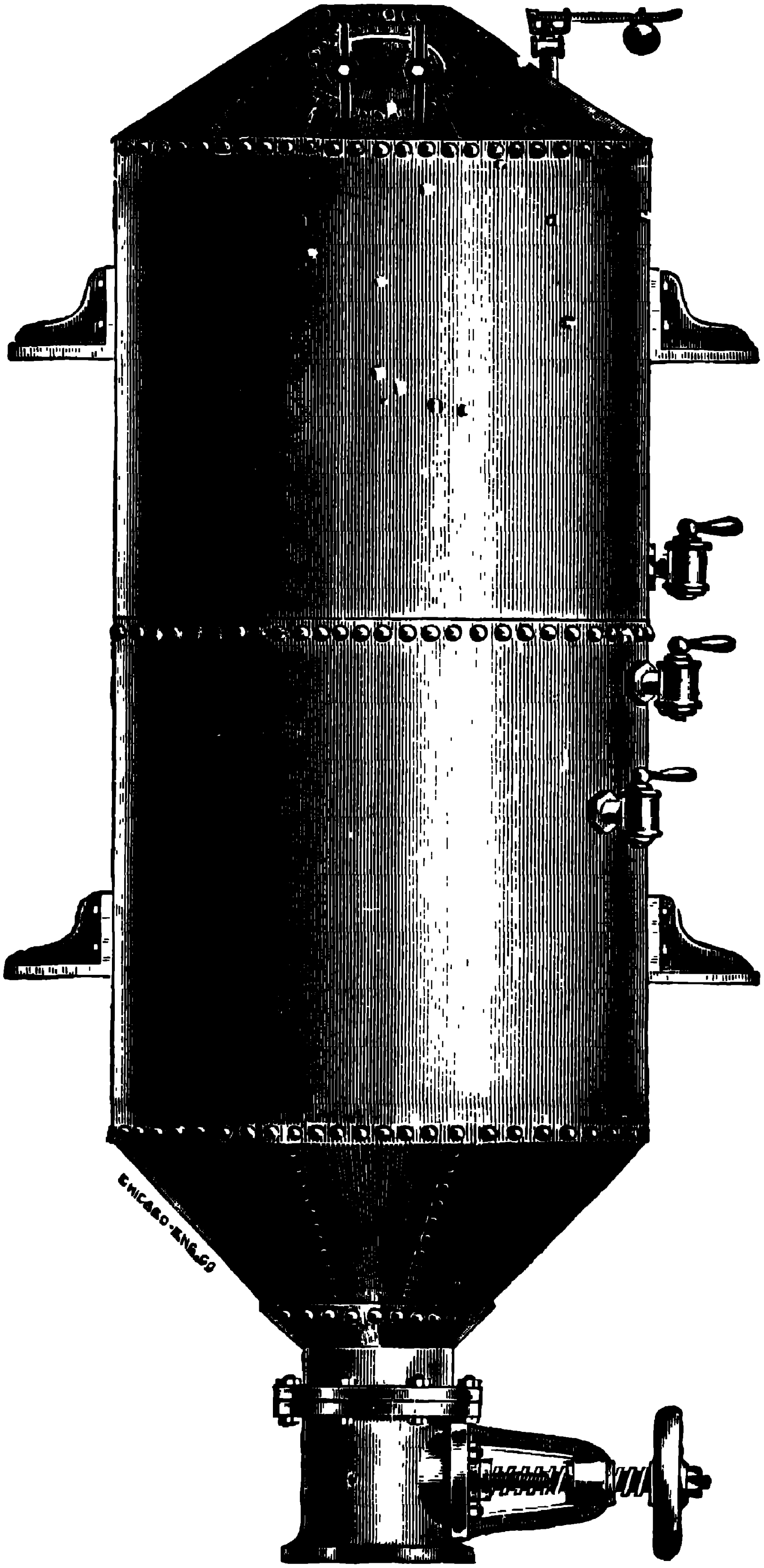

Fig. 9.—Packers' Rendering-tank

out of the vent-pipe, steam is turned off and the water blown out through a discharge-pipe for this purpose at the bottom. At the termination of the cooking period, steam is turned off and the contents of the tank allowed to settle in the order of their specific gravities. Steam is allowed to blow off through the vent-pipe. The rendered stock is drawn off first through the top draw-off cock and through the lower ones as the level in the tank subsides. Should the fat yet remain below the lowest discharge, water is admitted at the bottom of the tank and the fat thus raised and run out. The remaining contents of the tank are discharged, through the gate-valve at the bottom, into vats immediately below the rendering-tank, from which the liquor is allowed to run to a series of catch-basins to remove remaining traces of fat, and finally to the evaporator, usually a triple effect to be converted into "stick." The solid matter remaining is wrapped in cloths and subjected to great pressure to remove the moisture and fat that are yet retained. This dry-press cake is mixed with concentrated "soup" liquor or "stick" to form the tankage fertilizer of the packing-house.

The quality of the fat thus obtained depends upon the quality of the material from which it is rendered and the skill exercised during the rendering process. The various grades of animal fats are always marketable and naturally an effort is made to obtain the product in the best condition possible and from every source that promises a profit. We therefore find numerous grades of tallow and grease obtained from every part of the animal received in every condition at the rendering establishment.

To illustrate the variety of animal soap-stock on the American market and their relative quality and corresponding price on the day of quotation, there has been arranged the accompanying table, Table V, in which the differences in quality are clearly evident.

Hog-fat.—Lard is a general term applied to the fat of the slaughtered hog, irrespective of its anatomical origin, and separated from the tissues of the animal by the aid of heat. Lard is classified according to the material used and the method of rendering. According to method of rendering, lard is classified as kettle and steam. According to the material used there are three general grades of lard, viz., neutral, choice, and prime steam.

Two grades of neutral lard are made—one from the leaf, the other from the back fat of the hog. In the packing-plants the leaf fat is taken from the animal immediately after killing, hung on mounted racks, and wheeled into refrigerators to remove as quickly as possible all animal heat. It is next chopped finely or reduced to pulp by machinery and melted in jacketed kettles exactly similar to those used for oleo-oil. When the melting process is complete it is allowed to settle, the precipitation of the fibre being accelerated by the addition of salt, as in the case of oleo-oil. After the settling process the clear oil is siphoned to a receiving-tank and is tierced for

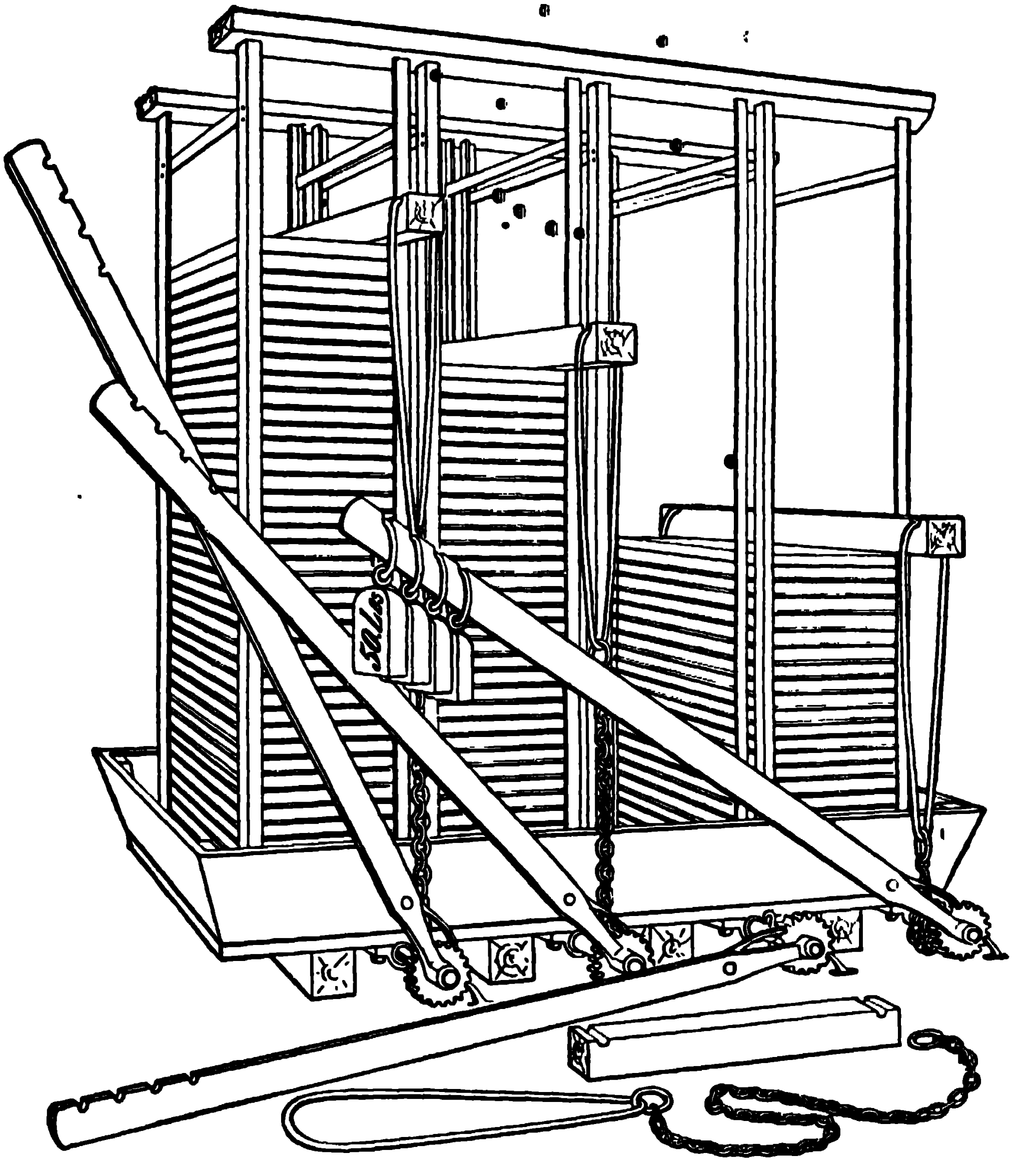

FIG. 10.—Lever Lard-press.

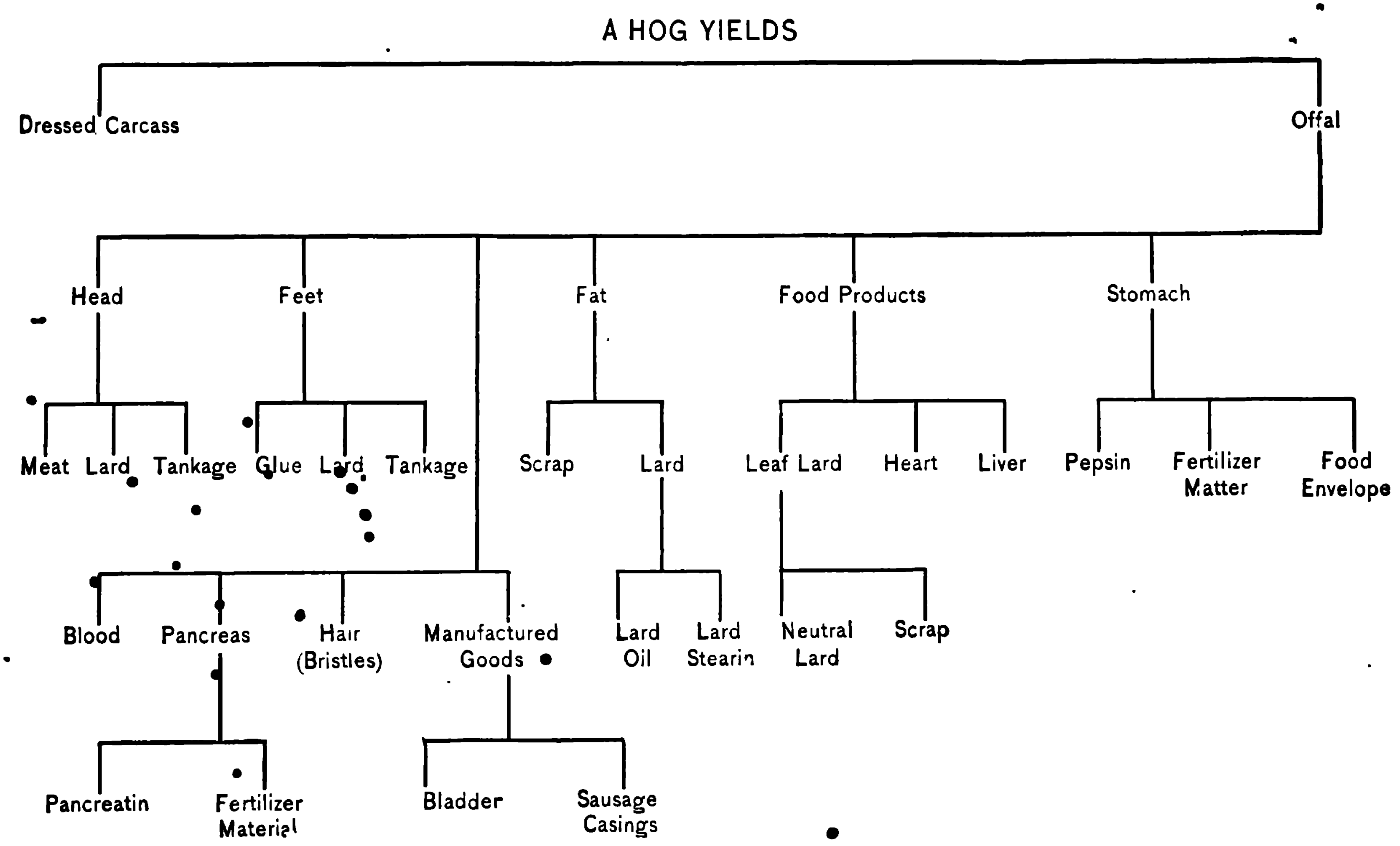

FIG. 11.—Diagram of Hog Products.

shipment. A good quality of leaf fat will produce by careful handling about 90 per cent of its weight in neutral, and each animal will yield an average of 8 or 9 pounds. Comparatively little neutral is made from back fat. The amount used, however, depends much on the relative demand for neutral and ordinary lard products, as it is sometimes more advantageous to work fats into one form than another. The oil made from back fat retains more of the flavor peculiar to lard and, like the lower grades of oleo-oil, is less free from stearin or other undesirable constituents. Some packing-houses mix a small per cent of back-fat with the "leaf" in making their highest grade of neutral.

Choice lard is made from leaf and trimmings only and may be either kettle- or steam-rendered. The process of steam-rendering for lard is the same as described for the production of tallow. Prime steam lard may be taken to represent the whole or part of the fat of the animal rendered by steam.

Lard-stearin and Lard-oil.—These products are obtained from lard in the same manner as oleo-stearin and oleo-oil are obtained from "butter" stock of beef-fat. The stearin is used in the manufacture of lard compound and the oil for illuminating purposes. Lard-stearin of non-edible quality is a soap-stock for certain grades of soap.

Classification of Animal Soap-stock.—Referring to Table V Prime Packers' and edible tallow may be considered as butter-stock and is rendered as previously described from the caul fat of beeves. Packers' No. 1 and No. 2 tallow may be considered as obtained chiefly from trimmings and portions of the viscera. City Renderers' No. 1, which stands second to edible tallow in quality, is probably kettle-rendered by butchers, or a superior quality of steam-rendered tallow. Country Renderers', although coming from outside of the city, is obtained from the same material as that available to the packer although less careful selection of stock and less care in rendering may have occurred. Country Renderers' No. 2 is an inferior quality of the preceding grade of tallow resulting from the use of inferior material. No. 2 Packers', the lowest grade of tallow, corresponds in quality to yellow grease. The term "grease" is applied to inferior products obtained from either the hog or beef or both,

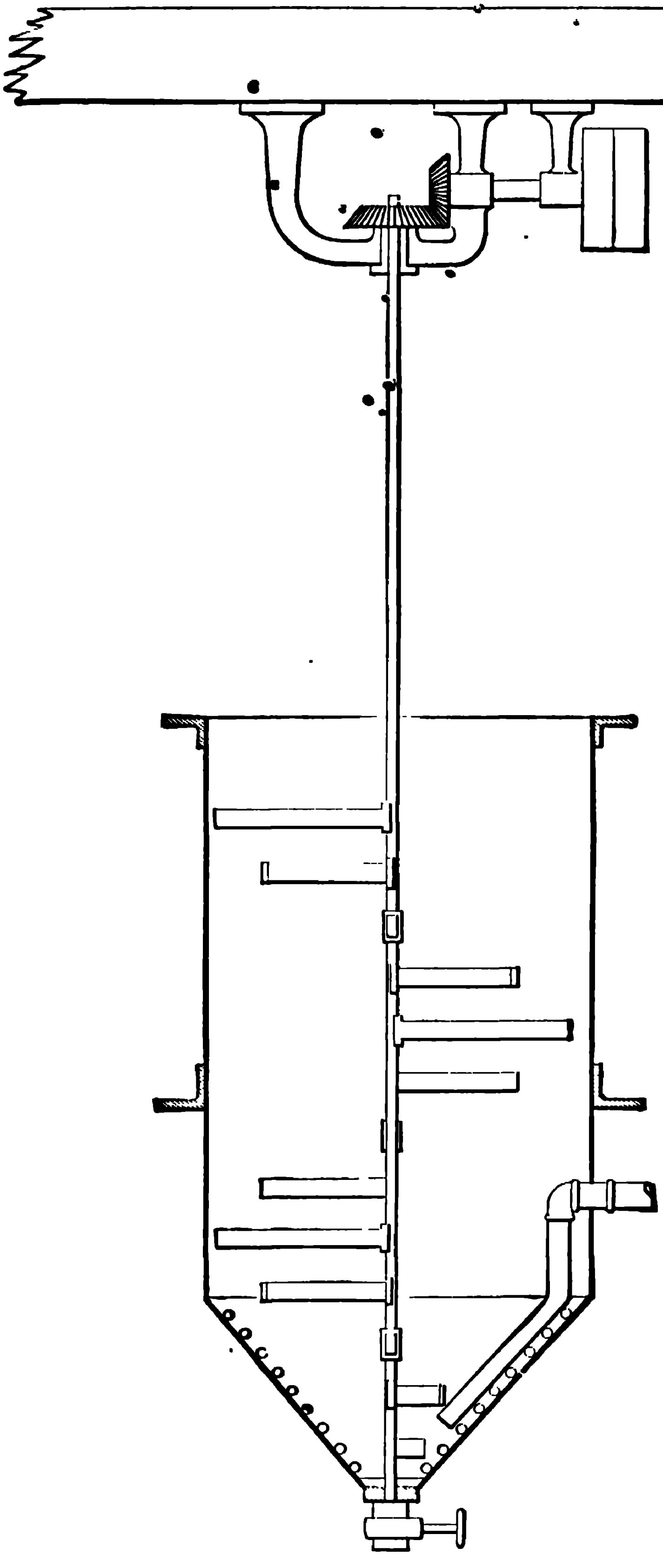

FIG. 12.—Lard-refining Kettle.

and likewise may be transformed into the corresponding stearin and oil. The color, odor, and body of such products are characteristic and indicative of their origin. A. White grease is steam-rendered from dead hogs (the intestines being removed) and from all other hog-stock not suitable for lard. B. White grease is an inferior quality of the preceding grade. Bone grease is steam-rendered from selected bones. Yellow grease is rendered from house, hotel, and restaurant collections. Brown grease is obtained from the same source and from the intestinal fats of dead hogs. The various grades of grease are rendered in the same manner as prime steam-rendered lard. As stearin is obtained by pressure from the corresponding fat from which it derives its name, the origin of the grades mentioned will be evident. By suitable treatment with bleaching agents it is possible to improve the appearance of any of the grades of soap-stock mentioned; the soap-making quality of the stock, however, is not thereby necessarily improved.

TABLE V.—QUOTATIONS OF ANIMAL SOAP-STOCK AT CHICAGO, SEPT. 15, 1898.

Soap Stock Arranged in Order Quoted.	Price. Cents.	Soap Stock Arranged in Order of Quality.	Price. Cents.
		Stearin:	
		Lard (city)	6 –6⅛
		Oleo (beef)	–5
		Grease:	
		White	–4
		Yellow	3 9/16–3⅝
Tallow:			
Prime packers' and edible	4⅛–4¼	Prime packers' and edible	4⅛–4¼
No. 1 (packers')	3⅜–3½	City Renderers', No. 1	3½–3⅝
No. 2 (packers')	2⅞–3¼	Country Renderers'	3½–3⅝
City Renderers', No. 1	3½–3⅝	No. 1 (packers')	3⅜–3½
Country Renderers'	3½–3⅝	No. 2, country	3 –3⅛
Country Renderers', No. 2	3 –3⅛	No. 2 (packers')	2⅞–3¼
Grease:			
Bone	3⅛–3¼	A. White	3½–3¾
Yellow	2⅞–3	B. White	3⅜–3½
A. White	3½–3¾	Bone	3⅛–3¼
B. White	3⅜–3½	Yellow	2⅞–3
Brown	2⅝–2¾	Brown	2⅝–2¾

It will be evident that no sharp distinction is possible in the classification of animal soap-stock. Stock from a generally inferior source may frequently be superior to the grade customarily assigned it. Although tallow and lard are respectively the fat of the slaughtered beef and hog, commercial grease may contain either or both of these fats. The presence of lard in tallow bought and sold as such is considered an adulteration.

In large cities animal soap-stock is now being extracted from sorted garbage by either the steam, sulphuric acid or naphtha-extraction process. It is generally known as recovered grease. It is used largely in the manufacture of candle-stock. Steam extraction of grease from garbage is the most common process and is carried out in the following manner:

The garbage, properly sorted, is elevated by conveyor to the charging door of the digesting tanks. These are upright steel tanks of 5 to 6 tons capacity and here the material is subjected to live steam for 6 to 8 hours, steam pressure being maintained at 30 pounds. When the matter has been thoroughly disintegrated, the envelopes enclosing the fat globules destroyed or softened and the bones so changed in physical property that they may be crushed between the fingers, the whole mass is dropped through a 12-inch valve into a box-like receiver. After settling, the free water and grease are run off by means of drop-pipes into separating vats and the remaining sludge elevated by bucket-pump into filter-cloths, after which it is pressed and sold as fertilizer.

In the weaving of woolen fabrics the raw wool must be first scoured and then oiled preparatory to spinning; the fabric is washed to remove the fatty and other bodies used in oiling the fibre and in the milling and falling process the cloth receives its final cleansing. In these several processes soap and other detergents and saponifiable oils are used in immense quantities. The recovery of this waste-oil and soap has reached a more advanced stage abroad than in this country, but the product is of too inferior a quality to recommend it for soap-making.

Red Oil.—Red oil is a commercial name given to crude oleic acid. This is obtained by the hydrolysis or saponification of a glyceride into its fatty acids; it is a by-product and of much less value than the substance manufactured, viz., stearic acid.

It can be obtained from many substances, principally from tallow and grease when these are employed in candlemaking. Its manufacture by various processes is described in detail in the section devoted to the manufacture of candle-stock.

In general the red oil thus produced is utilized by conversion into so-called "olein soap" by direct saturation with alkali, but various attempts have been made to employ it as a source of more valuable products; as, for instance, among others may be mentioned, (1) v. Schmidt, by the use of heat and zinc chloride forms isoleic acid and stearo lactone; (2) Radisson, by fusing with alkalis forms palmitic acid; (3) Zarar, by the action of chlorin, nascent hydrogen, water, and heat forms stearic acid.

Red oil is chiefly used for soap-making and as a wool oil. If made by the lime-saponification process it is called "red oil," saponified olein and saponification olein; if made by the acid saponification it is also termed distilled olein.

Red oil has, besides the names above mentioned, the following: Commercial oleic acid, Olein and Elain. In its pure state it is transparent and of a yellow to light-brown color; if turbid it has a dark-brown color. The former quality is termed "pale olein" (French, Oleine blonde; German, Blondes Elain); the dark variety is "red oil."

Red oil often contains a notable quantity of solid fatty acids, i.e., palmitic and stearic; if it has been obtained by acid saponification there may also be present more or less isoleic acid and hydrocarbons. Red oil contains a very variable amount of unsaponifiable matter, Allen giving as high as 10.3 per cent.

Red oil is also obtained by the distillation of "cottonseed foots" and from recovered grease; in the latter case the unsaponifiable matter is considerable, and this is used as a wool-oil and as inferior soap-stock.

Handling of Tallow in the Factory.—Probably the most vexatious problem that confronts the factory superintendent is the handling of tallow preparatory to its introduction into the soap-kettle. The most satisfactory method is one that combines cleanliness, convenience, and freedom from odor. The method in daily use in each factory represents the most practicable one attainable in this direction under the conditions there prevailing. It is possible, however, that a brief review of the various methods of handling tallow may enable those less advantageously situated in this respect to so modify their present method that these prime desiderata, viz., cleanliness, convenience, and freedom from odor, may be more fully attained.

Tallow in its various grades is received at the factory in two styles of containers, viz., barrels and tank-cars. Wherever the volume of business and location of the factory will warrant, the receipt of the tallow in tank-cars represents the maximum in cleanliness and convenience that at present seems to be possible. Tank-cars will hold as a rule about 40,000 pounds of stock, equivalent to over 100 barrels, which surpasses the capacity of a box car. When one considers that with suitable piping, pumps, scales, and storage-tanks a tank-car can be emptied in about an hour, the superiority of this method of receiving tallow over that involving the use of steam in emptying barrels requires no further comment.

Not every factory, however, has the convenience of track connections, thus being under the necessity of receiving all tallow in barrels. Tallow barrels are derelicts—not good enough to be used for other material, and as a result few are found that do not leak, especially in summer when the contents are more or less fluid, and are invariably soiled with the material they contain. To conserve cleanliness and to diminish time and labor required for handling, the receiving platform, scales, and steaming-tank should be near together. This condition can be attained by having the tallow received, stored, and emptied in a separate building provided with suitable steam- and tallow-pipe connections with the kettle-room. Ideal conditions are never met with in factory work, and no matter what effort is made to render offensive work less objectionable, much, as a rule, remains to be desired. It is true that the more barrel tallow is transferred from place to place in the factory the more difficult it will be to keep that part of the factory in a cleanly condition.

No two methods of disposing of barrel stock after it has been received are alike, each being determined by the conditions prevailing in each factory. The use of steam for emptying containers is general. Even this convenience involves great inconvenience, especially in cold weather. The following are the requirements of a good stock-steamer and its appurtenances: The steaming-tank supporting the barrels should be long enough to carry that number of barrels as should best facilitate the work of emptying. A dozen barrels can be emptied as quickly as two when once they are on. There should be a separate steam-pipe connection for each barrel, as one barrel may be emptied before another. The disposition of the tallow as it flows from the barrel is varied. The modern idea in factory work seems to be, wherever practicable, to elevate all material and allow it to descend by gravity through the various manufacturing processes to the shipping-room. Ventilation and the disposition of vapors should determine the location of the stock-steamer. This is a prime consideration, especially when the factory is located in a populous district and the stock used is not of prime quality. Where objection is made to offensive odor, some device must be employed whereby a repetition of complaint may be avoided. This may consist of a suitable duct carrying the vapors from the enclosed stock steaming room to the factory chimney, the movement of the vapors being effected by the natural draft of the chimney or aided by a fan. Where discharge into the open air is not allowed, this method may satisfy all requirements. In case it proves inadequate, recourse may be had to conducting the vapors to the fire-grate of the boiler and there consuming them. This, however, under average conditions is an extreme precaution and seldom if ever employed. Discharge of the vapor by forced draft through a spray of water may sufficiently remove all objectionable odor. The odor from stock steaming results from a simple distillation by steam of the volatile ingredients of impure stock. The odor is least with stock of good quality and when less steam and hence more time are used. It is most objectionable when the other extreme is reached.

Soap-stock of Vegetable Origin.—Olive-oil is the vegetable soap-stock that is historically the most interesting, but in the modern practice of soap manufacture it is of limited importance owing to its high cost. Cheaper supplies of vegetable oils are now available.

There are over 200 species of plants whose seeds are used in making oil for illumination, medicine, food, soap, and for lubricating machinery. A large proportion of these plants are natives of tropical regions, many of which will not thrive in colder climates. On the other hand, there are many plants which could be profitably grown in the United States for the oil contained in their seeds. A few such plants are now cultivated in this country, principally, however, for other purposes than the use of their seeds for oil, as in the well-known cases of cotton and peanuts.

The function of the oil in the plant economy is to form a reserve supply of nutriment for the growing plant during the period of germination and before it is able to absorb food material from the soil and air. Starch serves the same purpose in the seeds of cereals; and starch and sugar in the roots of biennial plants like the beet. For this purpose the amount of fat in plants is in inverse proportion to the amount of starch and sugar which they contain, ranging from 71 per cent in the edible portion of the pecan to about 2 per cent in barley. The amount of oil and starch, stored usually in the seed, and starch and sugar, stored usually in the root, is an exceedingly variable quantity, depending upon the climate, soil, method of cultivation, degree of maturity, etc. We are concerned primarily with the oil, the yield of which is determined not only by the conditions mentioned, but by the conditions of handling the seed or fruit and by the process of extraction.

Oil is obtained from seeds by first crushing and then pressing them in cloth bags, or by boiling them in water and skimming off the oil which rises to the surface, or by using some chemical solvent, such as carbon disulphide, which extracts the oil. The first method is that generally employed, although the chemical process is coming into use to some extent. Seeds are either pressed cold in mills constructed especially for that purpose, or heat is used to coagulate any albumen present and to render the oil more liquid. In many instances both cold and warm pressure is used, but in the case of the best medicinal or table oils no heat is employed.

The method of using solvents commonly yields a greater amount of oil than does pressure, but is open to objections. The crude oils obtained by pressure or extraction are refined by filtering and the use of chemicals.

The residue of the seeds after the oil is extracted is called "oil-cake," and is often of great value as a stock-food or fertilizer. It is composed of the woody fibre and mineral matter which the seed contained, a small per cent of unextracted oil, and, of more value than all else, the proteid or nitrogeneous constituents of the seed. This gives it especial value as cattle-food, while the high per cent of phosphoric acid and potash in addition to nitrogen makes it a most valuable fertilizer.

Any commercial oil that will admit of saponification is available as soap-stock. The use of vegetable oils for this purpose is determined primarily by their cost, their properties and character of the soap desired to be made. When the economic character of vegetable oils is taken into consideration, the number available as soap-stock is very limited and is determined by their relative cost compared with that of the various grades of tallow. The successful cultivation on an extensive scale of plants bearing oil-producing seed, other than the cotton and the flax, depends upon their cost of production as compared with that of cotton and flax. The vegetable soap-stock of different countries, therefore, varies with the different economic conditions. The chief vegetable soap-stock in this country and, we may state, in the world, is cottonseed oil.

Olive-oil.—Olive-oil is expressed from the pulp of the ripe fruit *Oleo europæa L.*, native to southern Europe. The tree has been cultivated in the Mediterranean countries from the earliest times whence its cultivation has spread to various countries, notably California, where similar climatic conditions prevail. An average yearly temperature somewhat higher than that required by the grape is essential to the best development of the tree and fruit. Successful cultivation is not possible in any region where the temperature often falls below 20° Fahr.; best results are obtained where the average temperature of the coldest month does not fall below 43° Fahr. The tree is characterized by meagre foliage and strong root system, requiring less water than most cultivated trees. It grows best in sandy or loam soils rich in lime. The fruit varies greatly in size with different varieties of the tree, with corresponding variation in the quantity of oil and size of the pit.

In the following table this variation is shown for a number of the common varieties cultivated in California.

In the Mediterranean countries the procedure of oil extraction has been but little improved in centuries. In recent years, however, and notably in those countries where olive culture is being introduced, greater care and more improved appliances are being employed. The variation in the quality of the oil arises from the condition of the fruit when pressed and to the diligence used in the process of extraction. The oil obtained from unripe fruit is of

FIG. 13.—Mission Olive of California (single olive natural size).

TABLE VI.—AVERAGE OF FULLY TESTED VARIETIES OF THE OLIVE GROWN IN CALIFORNIA.*

Variety.	Number of Samples Examined.	Size, Number of Olives Per Pound.	Pit, Per Cent.	Per Cent of Oil.		
				In Whole Fruit.	In Flesh.	In Pit, Referred to Whole Fruit.
Varieties fully tested:						
Mission.	112	111.6	17.2	17.56	22.51	.61
Nevadillo Blanco.	57	157.3	17.3	19.21	22.92	.99
Manzanillo.	38	106.5	14.7	16.94	19.73	.55
Redding Picholine.	42	398.2	23.0	16.18	20.83	1.52
Uvaria.	29	205.1	25.5	13.71	18.51	1.07
Rubra.	35	196.1	17.9	18.58	22.01	.75
Oblonga.	32	179.4	18.7	13.34	15.68	.85
Columbella.	25	114.6	16.6	15.59	19.54	.60
Pendulina.	22	157.1	13.7	18.63	21.36	.96

* Agr. Exp. Sta.; Univ. of Cal.

a greenish shade and has a rough peppery taste. Over-ripe olives yield a pale oil, deficient in flavor, which soon becomes rancid.

Olive Crushing.—The olives, received from the estate on which the mill is located, or from adjacent groves, are sorted. Storage is generally provided on an upper floor, where the olives are spread out until they can be crushed; but they are never allowed to remain longer than 24 hours. Olives, particularly if wet when brought in, become mildewed and the oil is thereby deteriorated. The olive-mill consists usually of a stone basin, non-absorbent —a silicious conglomerate rock is used—in which the upright circular stone is made to revolve. The olives are added to a hopper above the mill and are introduced into the mill as required. The charge varies with the capacity of the mill; for a charge of 10 bushels of olives, about one hour is required for crushing. The crushed mass, consisting of pulp and pits, is now transferred to circular flat bags, or mats, made of straw, about 2 feet in diameter.

The mats as filled are placed in a press, a type of which is shown in Fig. 7, until the latter is full. Cold water is poured upon the pile of mats to facilitate the flow of oil. Pressure is applied gradually at first and increased to the highest at the end. The product thus obtained is the first pressure, or virgin oil, and is of the best quality. A small quantity of oil of inferior quality is obtained by a second pressing. This is obtained from the same pulp by grinding it with hot water and pressing as before. If the second pressing is done promptly an oil of fairly good quality, though deficient in "body," may be obtained; but if delayed, the quality of the oil obtained is very inferior.

FIG 14.—Essential Apparatus of Olive-oil Manufacture.

The oil of the first and second pressing is clarified by a process of settling and filtration through raw cotton. By the most improved process, the turbidity of the fresh oil, which is due to the watery juice of the fruit and some pulpy matter, is removed in modern methods of procedure by the apparatus shown in Fig. 15. The freshly expressed oil is introduced through the funnel into the separator at the bottom, which is kept constantly full of water, added, as shown, below the oil-jet. The water and press-liquid are thus kept in constant agitation. This results in the rapid deposition of the heavy impurities and the equally rapid rise of the small, light oil-drops. The oil very quickly forms a layer on top and can be drawn off by means of a faucet appropriately placed. The apparatus is continuous in its operation, and the oil is obtained free from all the grosser impurities. It is still, however, very cloudy, owing to the presence of small, light particles of vegetable matter.

Ample settling in clean vessels and decantation are employed for the final clarification. The first settling is made in a funnel-shaped apparatus shown in Fig. 17, from which it is run through cotton wool into settling-tanks. About one month is generally required for settling in the first tank, after which it is racked off carefully into the second and so on until the required color and clearness are secured. Three rackings are usually sufficient, and if the process is properly carried out, the oil is as bright as can be obtained by the most effective method of filtration and

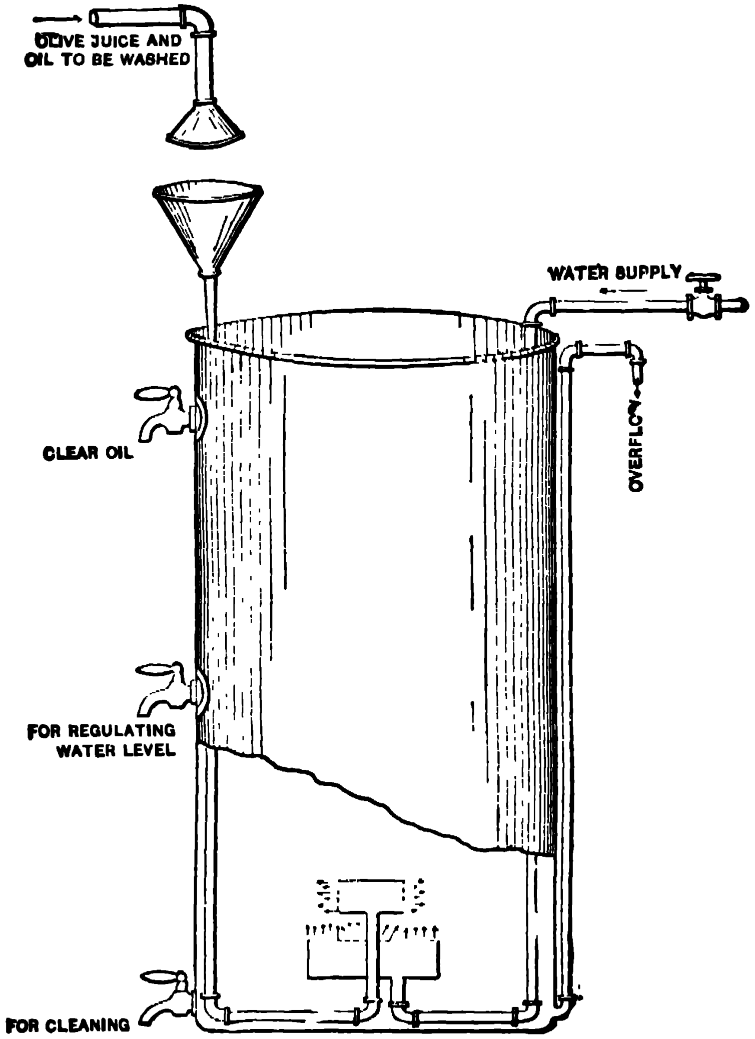

FIG. 15.—Separator for Olive-oil.

possesses the distinctive olive flavor and without the greasiness present in the filtered oil.

Olive-oil Foots.—The press-cake after the second pressing contains some oil, which may be extracted with some suitable solvent or by boiling up with water. By the extraction process the ground press-cake is mixed with a requisite amount of the solvent. The solution thus obtained is transferred to a closed vessel and heated to a temperature sufficient to expel the solvent by evaporation, which is condensed and recovered, leaving the oil behind. The solvents most commonly used are petroleum ether and carbon disulphide, the latter having the preference owing to its greater cheapness and safety.

The oil produced by chemical treatment of the press-cake is usually taken up by the soap trade, and its color and odor readily distinguish it from the oils obtained by the application of pressure. The oil obtained by the use of petroleum does not emit the disulphide odor, and it has a better color. The remaining pulp, or meal, after the extraction of the oil is immediately dried, in order to prevent fermentation, and sold in sacks for fertilizing purposes.

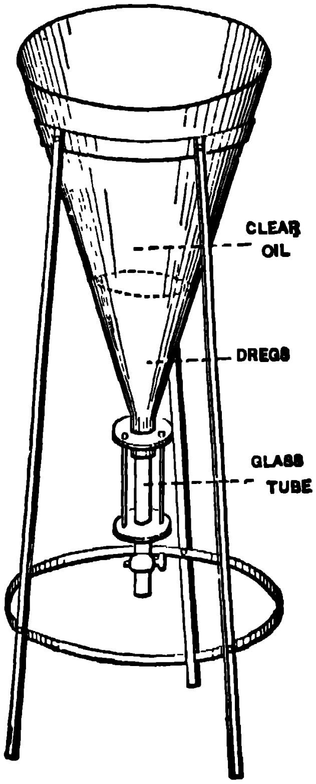

FIG. 17.—Clarifying Apparatus for Olive-oil.

By the water-extraction process the ground press-cake is agitated with hot water until the clean pits fall to the bottom of the tank, while the refuse pulp floats on the surface. The supernatant mixture of pulp, oil, and water is drawn off and the oil rising to the top is removed by skimming, while the pulp is again pressed and yields a little oil. This recovered oil is nearly black, and if extracted with solvents carries, to a greater or less degree, the characteristic odor of the solvent used. It is of less value than the oil extracted by the water process, but has the advantage in that every particle of the oil is recovered. The yield of oil from press-cake by the solvent-extraction process averages 10 per cent; by the water-extraction process only about one-half as much. The oil recovered as described, either alone or in admixture with inferior grades of the second pressed oil, comes into trade as "olive-oil foots." To distinguish it from the comestible oil it is called industrial olive-oil.

The industrial oils are purified by the use of sulphuric acid. The process consists in the agitation of the oil, into which has been poured 2 per cent of its weight of sulphuric acid at 66° Bé. and a quantity of water. The mixture is allowed to repose and is then decanted and filtered by ordinary processes. If the oil be heated to 60° or 70° C., the proportion of sulphuric acid may be reduced to as little as one-half of 1 per cent. If industrial oils are to be purified by the use of soda, the rule is to pour into the oil a weak alkaline lye, the liquid being then vigorously mixed. After repose the liquid forms in three strata—the alkaline solution appears at the bottom, the clarified oil at the top, and in the middle is a stratum in emulsion. The upper layer is removed, and to the middle layer is added another quantity of the alkaline solution, the product being again agitated and allowed to repose, whereupon it forms three strata as before. The operation is repeated until the bottom layer is but slightly turbid. The oil is then decanted and filtered after settling.

Linseed-oil.—Linseed-oil, the representative drying oil, is of use as a soap-stock only in the manufacture of soft soap, and for this purpose it finds but limited use in this country. In European countries, where it can compete successfully with other soap-stock it is used extensively for this purpose. The oil is expressed from the seed of the common flax (*Linum usitatissimum*) which is cultivated throughout the temperate zone, the chief sources of production being Russia, India, and the United States. Flax cultivation in the United States has moved westward with the development of farm-land and the territorial specialization of crops. The trend is now toward the northwest, and it is expected that within a few years the whole crop will be grown in Minnesota and the Dakotas. In this country the flax is cultivated chiefly for its seed.

The seeds are flattened, elliptical oval, pointed at the lower end, smooth, shining, and of different shades of brown. They are 3 to 4 mm. long, 2 to 3 mm wide, and about ½ mm. thick. They are produced in a ten-seeded globular capsule, which either remains closed at maturity or in some forms opens suddenly, scattering the seeds. Unlike cotton, flaxseed contains beneath the shell a hard layer of endosperm surrounding the embryo. This layer, however, is comparatively thin, and the oil is derived principally from the fleshy, oval, or narrowly heart-shaped seed-leaves (cotyledons) which it encloses. The outer layers of the seed-coat become transformed into a mucilage when moistened with water, which gives the seeds their principal medicinal value.

The seeds contain 30 to 35 per cent of oil, 20 to 28 per cent of which is obtained by pressure or extraction. Cold pressure yields 20 to 21 per cent, and the oil thus obtained is used in Russia and Poland as a substitute for lard and butter in cooking. It is of a pale yellow color, and has a rather pleasant taste and smell. The warm-pressed seeds give from 27 to 28 per cent of an amber-colored oil, which has a stronger and somewhat acrid taste. The oil from fresh flaxseed is sticky and turbid; hence, as a rule, seeds are pressed from two to six months old. Linseed-oil is rather thickly fluid, rapidly absorbing oxygen, and becoming thicker, then dry and hard, when exposed to the air. It therefore belongs to the group of drying oils, of which it is the most important.

The press-cake is used as a cattle-food. Linseed-oil is used chiefly in the manufacture of paints, varnishes, printer's ink, oil-cloth, etc.

Cottonseed-oil.*—Cottonseed-oil is the most valuable product of cottonseed which yield under the usual treatment the following average amount of products per ton of seed:

	Pounds.
Crude oil	300
Cake	750
Lint	20
Hulls	800
Loss or waste	120
Total	2000

For many years cottonseed were considered of use but to plant. Thousands of tons of this seed were annually burned or dumped into the rivers. But at last it was shown that cottonseed were not only valuable as a fertilizer and feeding-stuff in their crude state, but were capable of furnishing products that are among the most important elements in our national economy. The oil is the main product, and is used for a great variety of purposes. The residue after the removal of the oil is a valuable fertilizer and feeding-stuff, the hulls excellent fuel, the ashes of the hulls a fertilizer rich in potash and phosphoric acid, and the refuse from the oil refining valuable stock for the manufacture of soaps. Thus a vast mass of material which was once looked upon as necessarily a waste product has become of very great commercial value.

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* See "Cottonseed Products," by L. L. Lamborn.

As it comes from the gin, upland cottonseed consist of the seed proper and the soft down which adheres closely to the seed-hulls. This down, known as "linters" to distinguish it from the longer fibres which constitute the lint of commerce, does not need to be removed from that portion of the seed which is reserved for planting, although delinting, besides yielding a valuable commercial product (linters), makes the seed easier to handle and retards fermentation.

As received from the gin the seed are mixed with more or less foreign matter constituting the waste, which it is necessary to separate. The seed are first passed through a reel, or revolving screen of wire netting or perforated metal, whereby sand, cotton-bolls, sticks, etc., are removed. Cleaning seed should be a thorough process, otherwise any foreign matter, as fragments of iron, allowed to remain may injure machinery used in subsequent stages. From the sand- and boll-screen the seed pass to the delinters, which are similar in construction to the ordinary cotton-gin and serve to remove the "linters" previously described. From the delinters the seed pass to the hullers, which hull or decorticate the seed. By use of a screen similar to the sand- and boll-screen, the meats and hulls are separated. The meats are now conveyed to the crushers, which serve to reduce them to flakes, whereby the oil-cells are ruptured and the mass reduced to a uniform consistency.

After this crushing the meats drop into a conveyor, which delivers them to the heaters. These are large cast-iron steam-jacketed kettles provided with stirrers which keep the meats moving while they are being cooked. The duration of the cooking varies from twenty to thirty minutes, according to the condition of the kernels and the good judgment of the cook, a human quality here called for the first time to supplement the automatic mechanism that has conducted the seed to this point through all the various processes it has undergone in its journey from the seed-house. The object of the cooking is to expand the oil in the meats and render it more fluid, to coagulate the albumen, and to drive off the water, which not only reduces the quality of the oil, but is liable to work serious injury to the expensive cloths used to envelop the cakes in the press. Very dry meats may sometimes be cooked in twelve to eighteen minutes, while fresh seeds may require forty-five minutes. Close to the heaters stands the "former," which shapes the meats into cakes for the press. The cakes as they come from the former are wrapped in hair-cloth and removed by hand to the press, where they are arranged in a series of boxes, one above the other, between the plates of the press, and subjected to a pressure of 3000 to 4000 pounds to the square inch by hydraulic power. The cakes, pressed as solid as boards, are taken from the press, stripped of the cloths, and stacked to dry. When dry they are passed through a cake-cracker, which breaks them into fragments of a size suitable to be fed to a mill. The mill grinds these fragments into a fine meal, which is put up into sacks containing 100 pounds. Sometimes the meal is bolted to separate it from small pieces of the hull, which, being tough and leathery, are not readily ground up.

The crude oil as it flows from the press is conducted to storage-tanks or to the filter-press, where foreign matter, chiefly meal, is separated by sedimentation or filtration.

Crude cottonseed-oil is a thickly fluid, deep red to almost black oil, the quality of which is determined by the quality of the seed and the skill used in their manipulation. It is contaminated by moisture, albuminous and mucilaginous matter of the seed and by coloring matter. The value of the oil is determined by the loss on refining which loss includes, besides the depreciation in volume from the separation of the non-oleoginous matter just mentioned, the separation of the free fatty acids of the oil. Commercial caustic soda, 74°, is the common refining agent, and is used in solu-

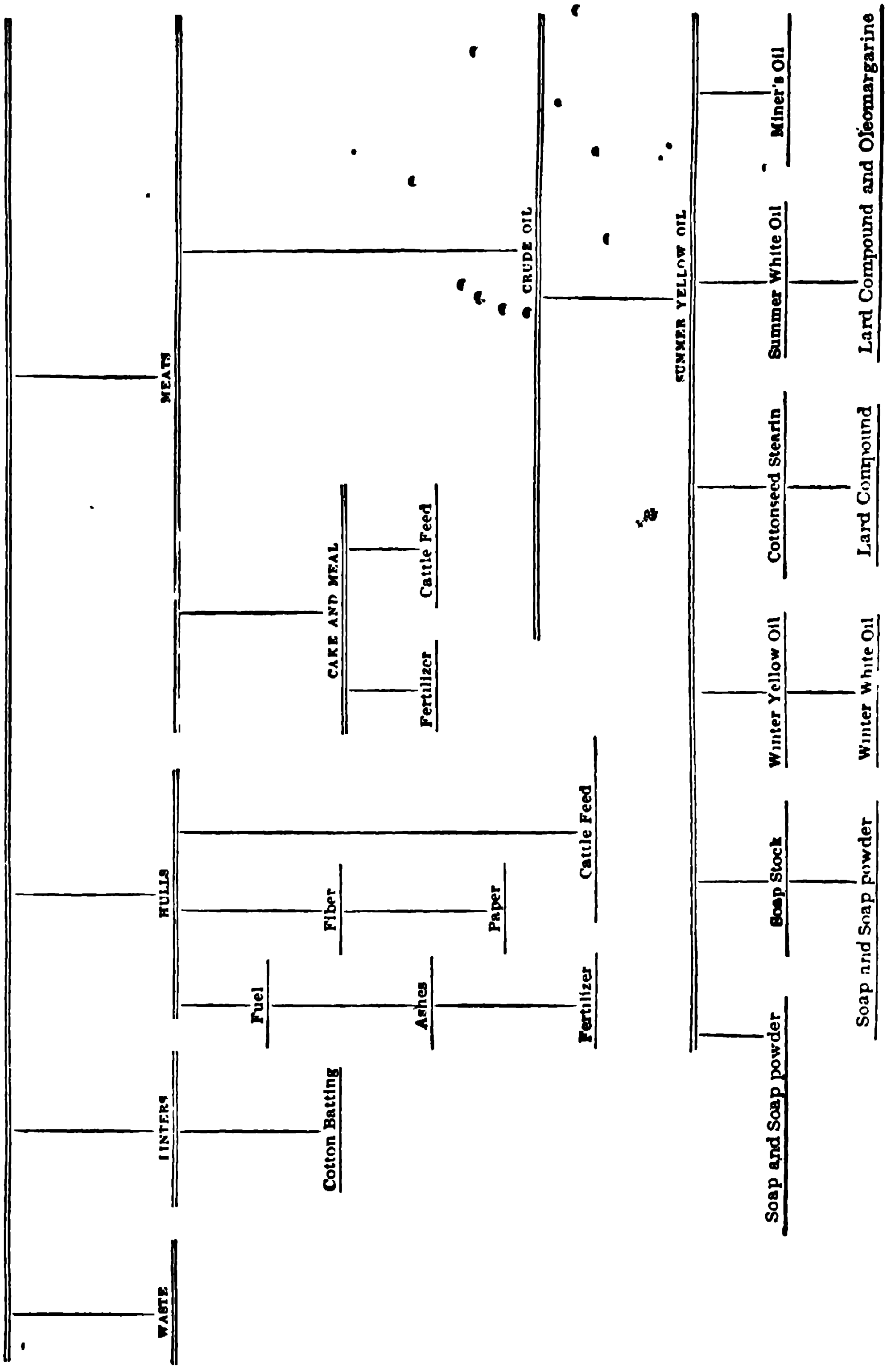

FIG. 18.—Diagram Showing Products Obtainable from Cottonseed.

tions of varying densities and proportions, according to the character of the crude oil.

Refining consists in agitating by an air-blast in a kettle of about 125 barrels capacity, a weighed quantity of the oil with the caustic lye, the density and proportion of which having been previously determined by a refining test on a sample of the crude oil to be refined. The procedure of refining is as follows: The crude oil to be refined should be weighed accurately and a sample taken from the same for the refining test, the crude oil in the meantime having been transferred to the refining kettle which has previously been thoroughly cleaned. In pipe connections and construction the refining kettle is similar to the ordinary soap kettle. The oil then should be heated to 85° Fahr. and the caustic-soda solution of required quantity and strength is added, meanwhile keeping up vigorous agitation. The more quickly the lye is added the better. Care should be taken while adding the lye that it is uniformly distributed over the surface of the oil. For this purpose a perforated criss-cross pipe supported over the surface of the oil is used. After the lye has been added, the mixture should be agitated vigorously for about ten minutes. Then apply a gentle heat, through the medium of an open steam-coil, heating to about 125° Fahr. While doing so agitate the oil gently. The time from the addition of the caustic-soda solution to the attainment of the maximum temperature, viz., 125° Fahr., should be at least fifteen minutes. There is no objection, however, to taking longer time than this. Agitation should be continued gently, until there is a disposition on the part of the impurities to curdle. Then agitation should be discontinued and the contents of the kettle allowed to stand until the impurities subside. The sediment consists of the coagulated albuminous and muscilaginous matters of the oil, with much of the coloring matter, in intimate admixture with soda soap of oleic acid, together with that portion of the caustic-soda solution added in excess. This material is the raw material of cottonseed-oil soap-stock. After sedimentation has been effected and the oil cooled to a satisfactory temperature, the clear supernatant yellow oil is siphoned off by means of the swing joint-pipe to the finishing kettle where it is washed free of alkali at a temperature not exceed-

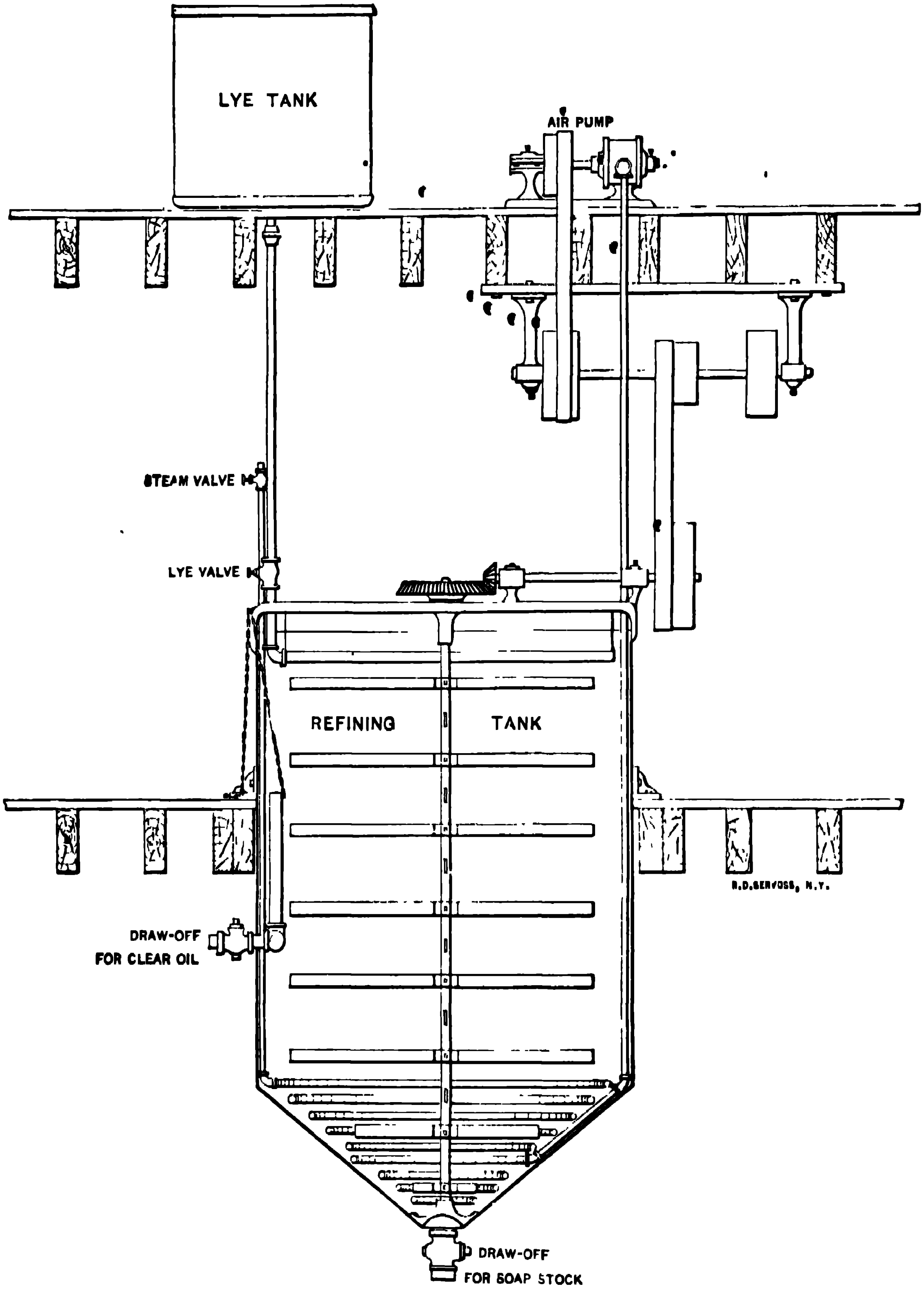

FIG. 19.—Apparatus for Refining Cottonseed-oil.

ing 100° to 105° Fahr. After the first washing, the wash-water is withdrawn from the bottom of the kettle until oil comes; then fresh water should be added and while adding the water the oil should be kept in thorough agitation by means of the air-blast. The water should not be colder than 100°–105° Fahr. After all traces of caustic soda have been separated by washing and the water settled out, moisture should be further abstracted by applying steam to the closed coils and heating to a temperature not exceeding 125° Fahr., agitating all the time. Should difficulty be experienced in removing the last traces of water, the addition and thorough admixture of powdered plaster of Paris is recommended, the amount used to be determined by the amount of water retained by the oil. The determination is best made by taking small samples of the oil and treating it with the plaster of Paris and filtering. After agitation with the plaster of Paris has been conducted for a sufficient length of time, the oil is filter-pressed. Oil flowing from the filter-press, after the foregoing procedure has been followed, should be ready for barrelling as prime summer yellow oil when made from pride crude.

According to the rules adopted for the government of transactions in cotton-seed products, cotton-seed, crude oil and refined oil are classified and graded as follows:

Cottonseed shall be divided into two classes: Prime seed and off seed.

(1) Prime seed shall be clean, dry, sound seed, free from dirt, trash and bolls.

(2) Off seed—Seed not coming up to the requirements of prime seed shall be considered off seed. Off or damaged seed shall be settled for on its merits and comparative value as against the value of standard prime seed.

Crude cottonseed-oil shall be classed and graded as follows: Choice crude oil must be made from sound decorticated seed; must be sweet in flavor and odor, light in color, free from water and settlings, and test not over 1 per cent of free fatty acids; shall produce, when properly refined, choice summer yellow oil, at a loss in weight not exceeding 6 per cent for Texas oil, and at a normal loss for oil from all other parts of the country.

Prime crude cottonseed-oil to pass as prime must be made from sound decorticated seed, must be sweet in flavor and odor, free from water and settlings, and must produce prime summer yellow grade by the usual refining methods, with a normal loss in weight, provided the oil shall not be rejected for a nominal amount of settlings; but reasonable reduction shall be made in value for all such settlings in excess of one-quarter of 1 per cent of free fatty acids, not to exceed 1½ per cent.

Off Oil—All oil neither choice nor prime shall be called "off" oil, and shall be sold by sample.

Refined cottonseed-oil shall be classed and graded as follows, summer yellow only being considered.

Choice must be sweet in flavor and odor, of light straw color, clear and brilliant in appearance, free from moisture and must bleach to a choice white.

Prime must be clear, sweet in flavor and odor, and of yellow color, not reddish, free from water and settlings.

Off to be classed as off refined: All oils having any objectionable flavor or odor. Off yellow oil to be of a yellow and not a reddish color.

A tank (tank car) of cottonseed-oil for contract purposes shall be 125 barrels. A barrel of oil, if sold loose, is fifty gallons. A gallon of oil is seven and a half pounds avoirdupois.

Crude cottonseed-oil may be sold either loose or in barrels, as agreed between seller and buyer. If in barrels, they shall be good, iron-bound barrels, properly silicated, or thoroughly steamed and cleaned refined petroleum barrels. Packages must be in good shipping order, and contain not less than forty-eight gallons each.

All summer yellow oil not graded as "off" is the raw material for the preparation of edible products. Off summer yellow oil is the grade used in soap-manufacture.

Cottonseed-oil Soap-stock.—After the removal, through the swing-joint pipe of the refining-kettle, of the clear, supernatant yellow oil, the sediment in the bottom, consisting of the non-oleaginous matter of the crude oil, together with the soda-soap of oleic acid mixed with uncombined oil and caustic soda-solution, may be transferred to a special kettle or tank reserved for this purpose where it is heated up, and the free oil rising to the surface is skimmed off and returned to the crude-oil tank. The preparation of marketable soap-stock from this material comprises a series of salt-washes to discharge the coloring matter. The method of treatment depends upon the utilization of the material. If the soap-stock is to be worked into soap or soap-powder at the place of its production, the procedure followed is a part of the kettle-room practice. If it is to be marketed at once as soap-stock the procedure followed as stated before comprises washing with brine under suitable conditions to discharge the organic impurities and coloring matter. After the removal of the greater part of the uncombined oil by heating up the mass, settling and skimming, as already described, the residual mass is transferred to the soap-kettle where it may be allowed to accumulate. Unrefined cottonseed-oil soap-stock is analogous to the nigre of soap-manufacture and its treatment is along the same general lines as that to which the nigre is subjected. In boiling up the soap-stock the excess of caustic soda, which settled out in the refining kettle in admixture with the organic sediment, is absorbed by the free oil. There remains a soap mixed with more or less free oil, and highly contaminated with organic impurities. The soap is grained sharply with dry salt and the very foul lye which is separated is discharged into the sewer. The number of salt-washes to which the soap is subjected depends upon the degree to which it is contaminated and upon the color desired in the finished product. After withdrawing the wash-water, fresh water is added, with constant boiling, until the soap just closes. Dry salt is again added to produce the desired grain and the separated pickle containing the discharged impurities is withdrawn as before.

The impurities may also be discharged by settling the soap, whereby a nigre is formed, which while it contains some soap, also contains practically all of the organic impurities. Settling the soap for this purpose may either precede or succeed a series of salt-washes. If the soap is settled at once, after the contents of the kettle have stood sufficiently long to form a nigre, the latter may be run into the sewer until good soap comes. The soap thus purified may be either barrelled at once, or subjected to a pickle-wash, after which it is barely brought to a close and then barrelled.

According to rules governing transactions in cottonseed products, all sales of soap-stock, unless otherwise agreed upon between buyer and seller, are on a basis of 50 per cent of fatty acids, not to fall below 40 per cent. If containing less than 40 per cent, soap-stock is not considered merchantable. As will be clear from its method of production, soap-makers' yield does not apply to cottonseed-oil soap-stock. The yield from this stock depends upon the percentage of fatty acids, the greater this per cent the lower as a rule is the degree of hydration, and upon the percentage of free or uncombined oil.

The color of cottonseed-oil soap stock varies from yellow to brown according to its degree of purification, which treatment likewise determines the odor. Both color and odor are characteristic and betray the origin of the stock in about all detergents containing it as an ingredient.

Corn-oil.—In addition to the use of maize or Indian corn as a food, in the raw state for animals, and ground, as corn meal, for man, it is the raw material of four important manufactured products, viz., whiskey, alcohol, glucose, and starch, in all of which the embryo, or germ of the corn kernel is a by product. The grain of corn consists of an exterior covering, or hull, enclosing starch which in turns surrounds the embryo, or germ. This germ contains the oil, in which it is present to the extent of about 35 per cent. The average composition of maize or Indian corn is as follows:

	Per cent.
Oil	5.20
Carbohydrates (starch therein 54.8 per cent)	71.22
Albuminous substances	10.46
Ash	1.52
Water	11.60
Total	100.00

The chief and at present the only commercial source of corn-oil is the starch and glucose industries, in which it is obtained in the following manner: The shelled corn is first steeped in water at 150° Fahr. for three to five days, whereby the hull is loosened by swelling of the grain. Sulphurous acid is added to the water to check fermentation. The softened kernels are next crushed between rollers, whereby the hulls are separated from the starch and germs which remain whole and of about the size of a grain of rice. By means of a "degerminator" the broken corn is separated into starch and hulls and germs in virtue of their different densities. The germs float upon the surface of the water and are removed and dried. The dried germs are then ground or crushed, cooked and formed into cakes which are then subjected to pressure in a hydraulic press, after the manner of cottonseed-oil extraction. The crude oil is dark colored, being heavily charged with albuminous matter. On refining it yields a bright yellow oil which may be further refined to the almost complete absence of color. It possesses drying properties sufficient to recommend it as a paint oil for special purposes. The odor is characteristic and more persistent than that of cottonseed-oil. The source of corn-oil is abundant. The extent of its use and its commercial position among the seed-oils of the United States will be determined by competition with cottonseed-oil.

Cocoanut-oil.—Cocoanut-oil appears in the markets of the temperate zone as a soft white fat of characteristic taste and odor. In tropical countries it appears as an almost water-white, rather turbid, oil. Quotations are for three grades, viz., Cochin, Ceylon, and Coprah-oils. The terms Cochin and Ceylon refer primarily to the geographical origin of the fruit and do not strictly indicate relative quality. Coprah is the sun or kiln-dried pulp of the fruit; the same term is applied to oil expressed from it. It is of the lowest quality. It has been suggested that the relative quality be indicated by the terms "white," "prime," and "yellow," instead of, as at present, by the territorial designation stated above. Cochin cocoanut-oil is considered generally as of superior quality, although Ceylon oil of equal quality is frequently received; also Cochin oil is often of the grade commonly assigned Ceylon. Cochin cocoanut-oil is obtained from fruit grown in the province of Cochin China and adjacent territory and the Philippines. Ceylon oil is obtained from the fruit grown on the island of Ceylon. Coprah-oil may be of either geographical origin. Both the dried fruit and the oil are important products of the Philippine Islands. In Cuba the industry was at one time in a flourishing condition and it is expected that with renewed attention the American demand may be supplied from this source.

The cocoanut is the fruit of the cocoa-palm of which thirty species have been discovered and classified. With each variety the fruit varies in size, weight, shape and in the percentage of fibre, flesh, etc. The chief species are the *Cocus nucifera* and the *Cocus butyracea* which furnish the greater part of the cocoanut-oil of commerce. The cocoa-palm is native to the tropical zone and is cultivated extensively in Central and South America and West Indies, but it is in Asiatic countries that the tree flourishes most luxuriantly and from which our chief supplies are obtained. Botanically the fruit is a drupe, generally ovoid in shape and as large as a man's head. The shell, or endocarp, is bony, is pierced at the base with three holes and encloses at maturity the white compact pulp from which the oil is expressed. What we are familiar with as the cocoanut is covered in nature with a fibrous mass from which mats, ropes, carpets, brushes, etc., are made by the natives. It is estimated that the kernels of 500 nuts give an average production of one hundredweight of oil, and the kernels of 240 nuts produce about one hundredweight of coprah. Reduction of the fresh fruit and its compression to separate the oil are carried out on the same general lines as are employed in the manufacture of all seed-oils, although in regions where modern oil-mill machinery has not been introduced, the most primitive methods are used. As a result of its peculiar chemical composition, cocoanut-oil quickly becomes rancid on exposure. In regions deficient in shipping facilities, or where the fruit is intended for export for oil-expression, it is customary to break the bony envelope and allow the pulp to dry either spontaneously in the sun or by artificial heat. This product is the coprah of commerce. From its mode of preparation an inferior quality of oil is naturally to be expected.

The average free acidity of a number of lots of coprah-oil was found to be 8.40 per cent, while a similar average for Ceylon oil was 7.15 per cent.

The superiority of Cochin cocoanut-oil over Ceylon oil is due to the superior whiteness and quality generally of the Cochin coprah as compared with Ceylon coprah.

It is not supposed that the Cochin cocoanuts are better than Ceylon nuts, but that the superiority of the Cochin oil may be attributed solely to the better climate, and to the superiority of the preparation of the coprah from which the oil is made.

Cocoanut-oil is rarely adulterated, any sophistication being easy of detection. Refined cocoanut oil is used to some extent as a cooking oil. It is also used to some extent as raw material for candle-stock. Its chief consumptive outlet is the soap industry in which in the United States it is used in large quantities for floating, milled toilet and certain high-grade laundry and scouring (sand) soaps.

Palm-oil.—Palm-oil is expressed from the pericarp or fleshy portion of the fruit of the *Elæis guineænsis* and *Elæis Melanococca*, native along the west or gold coast of Africa. Here the palm grows in immense forests where for centuries the fruit has rotted unused upon the ground. The tree has never been made the subject of systematic cultivation. It begins to bear in its fourth or fifth year, increasing its yield until the fifteenth and continues to bear for at least sixty years. The fruit is about the size of a small plum and grows in thickly clustered bunches. A single tree will bear from 4 to 7 bunches each year. The fleshy portion of the fruit is the source of palm-oil. The kernels are shipped chiefly to the port of Hamburg where they are expressed for palm-kernel oil. They constitute the most important article of commerce of West Africa and of the German colonies in particular.

As the percentage of oil in the nuts varies with the conditions of growth, the yield of oil likewise varies from three gallons per year per tree in moist soil to one gallon per year per tree in dry soil. Three varieties of the tree are distinguished, having respectively orange, red, and dark colored nuts. The first yields the finest oil but small kernels; the second and third less oil but larger kernels. When the bunches of nuts are ripe they are collected and thrown into a hole in the ground until a sufficient quantity has accumulated to be made into oil. During this time the nuts undergo fermentation, producing "hard" oil. Freshly expressed nuts yield "soft" oil which brings a higher price in the European markets. This empirical classification arises from the different amounts of free fatty acids, due to the decomposition of the glycc ride, in the two grades of oil. The quality of the "hard" oil is also deteriorated by the presence of dirt, to remove which little care is taken. The oil is extracted by pressure and by boiling, the latter process being more commonly used. The nuts are boiled until the fibre is softened. They are then heaped in stone troughs and beaten with sticks until the fibre is loosened. The mass is then covered with plantain leaves and left for twelve hours, during which time heat is developed and a quantity of oil runs off. The nuts are then washed in hot water and the fibre separated and squeezed by hand.. The oil is finally freed from water and impurities by settling and boiling. This process is defective at every stage and cannot but produce those results that characterize commercial palm-oil. The best oil is produced from fresh and ripe nuts. They should not be allowed to ferment as the oil is thereby darkened and caused to harden through the liberation of free fatty acids. The final boiling further darkens the oil.

Fresh palm-oil has the consistency of butter, has a dark yellow to yellowish red color and a violet-like odor, with a sweetish taste. As it readily turns rancid on exposure, while its color at the same time becomes lighter, it is rarely received in fresh condition. With its varying degree of rancidity the melting point also rises or falls. The causes which render palm-oil liable to become rancid more rapidly than any other fat are to be found in the admixture of several readily changeable constituents of the flesh of the fruit. Lagos-oil is the purest known. For every ton of palm-oil there should be 2½ tons of clean palm-kernels.

Palm-kernel Oil.—In its chemical composition and soap-making properties, palm-kernel oil is closely allied to cocoanut-oil. Hamburg is the chief European port of entry for palm-kernels. The shells are removed from the seed and the oil extracted from the latter by processes analogous to those used in the manufacture of cottonseed, linseed, and corn-oils. The products thus obtained are palm-kernel oil and palm-kernel cake which has a high nutritive value for cattle-feeding. The oil is of varying consistency according to the temperature, of white to yellow color and of agreeable odor. It soon becomes rancid on exposure.

Peanut-oil.—The earthnut, groundnut, goober, pindar or peanut (*Arachis hypogœa*), as it is variously called, is a low, somewhat creeping annual belonging to the bean family. It is native to the tropics and has been cultivated extensively in Africa, India, the West Indies, and the southeastern United States. Only the lowest flowers bear fruit, which after blossoming lengthen their stems which penetrate the earth for several inches, where the fruit ripens. The fruit is 2 to 3 cm. long and 1 to 1.5 cm. thick with a furrowed yellowish pod, which contains from one to four seeds, one to two being the common number. In the United States peatnuts are usually planted after corn, 2 bushels of seed being used to the acre. A sandy loam containing some lime is the best soil. The crop is from 80 to 120 bushels per acre. The expression of oil from peanuts does not constitute an industry of any note in the United States. Cottonseed-oil machinery may be used for peanuts. Marseilles is the peanut-oil centre of the world for which supplies are drawn chiefly from India and East and West Africa. In the manufacture of the oil the pod and husk are first removed mechanically, after which the kernels are crushed and cooked preparatory to pressing, which is done in the same manner as with cottonseed; although the cake is usually reground and subjected to a second compression. The same processes of purification are applied to crude peanut as to other seed oils.

The average yield of oil for decorticated nuts at Marseilles of the crop of 1899 was as follows:

Description.	First Pressure, Per Cent.	Second Pressure, Per Cent.
Bombay nuts.	28-30	10-12
Mozambique nuts.	30 32	10.12
Senegal nuts.	20-22	10

The press-cake is ground for cattle-food.

In the face of competition from cottonseed and other sources, it is not probable that the peanut-oil industry will ever flourish in the United States. With the peanut the oil is the chief product and unless cost of production of the raw material can be very considerably reduced, it cannot compete successfully with a by-product. The chief consumptive outlet of the peanut in this country is as a food for which upwards of $10,000,000 are expended annually. That it possesses high nutritive value is shown by the following analyses:

TABLE VII.—ANALYSES OF PEANUTS.

	Number of Analyses.	Refuse.	Water.	Protein.	Fat.	Total Carbohydrates (Including Fibre)	Fibre.	Ash.
Peanuts:								
Edible portion—								
Minimum	4	......	4.9	19.5	32.3	15.3	2.0	1.9
Maximum	4	.....	13.2	29.1	48.8	40.4	3.0	2.4
Average	4	......	9.2	25.8	38.6	24.4	2.5	2.0
As purchased	. .	24.5	6.9	19.5	29.1	18.5	. . .	1.5
Peanut-butter, as purchased	2	.....	2.1	29.3	46.5	17.1	.....	5.0

The oil is regarded as equal to olive-oil, and it may be employed for every purpose to which that oil is employed. This oil forms from 30 to 50 per cent, by weight, of the shelled peanut; it has an agreeable taste and smell, and is more limpid than olive-oil, which it very much resembles. It is sweet, palatable and clear, and, in fact, great quantities are used, unknown to the consumer, instead of olive-oil. In India, Europe, Brazil and this country it is used medicinally in place of olive-oil, and it is also employed by manufacturers as a substitute for the latter in fulling cloth. As a lighting fluid it lasts a long time, but does not give as clear a light as other burning oils. It is a non-drying oil of a light straw color. It is used to a large extent in Europe for edible and industrial purposes, chiefly soap manufacture.

Peanut-oil soap is the basis of the famous Marseilles white soap. Cottonseed-oil is used only in default of peanut-oil or when the price is against the latter.

Castor-oil.—Castor-oil is obtained from seed of the castor-bean (*Ricinus communis*), a member of the family Euphorbiaceæ which furnishes over twenty species of oil-producing plants, most of them indigenous to tropical countries. The castor-bean is a native of India, but is cultivated in many parts of the globe.

The seed of the common large-seeded variety are oval, smooth, and shining, of a gray ground color, irregularly marked with brown. They are 10 to 20 mm. long, 6 to 10 mm. broad, and about 6 mm. thick, slightly pointed at the upper end, which is provided with a whitish, fleshy excrescence (caruncle). They are contained in a three-lobed, spiny capsule, each lobe containing one seed. When ripe, the capsules split from the bottom upward, throwing the seeds to a considerable distance. The kernel is composed of two thick, fleshy, white lobes of endosperm, which inclose a thin, leaf-like embryo. A small seeded form is used for medicinal purposes, while the large-seeded variety furnishes an oil used for lighting and in the making of soaps.

The shelled seed yield from 50 to 60 per cent of oil, which is more than that yielded by almost any other plant. The oil is obtained by pressing twice cold and a third time warm, by boiling with water and extraction by the agency of alcohol. It soon becomes rancid upon exposure to the air. The oil is extensively used in medicine as a purgative. Also in pomades, for illumination, soap-making, for lubricating machinery and in veterinary practice.

Castor-oil is the thickest and heaviest of the oils, its specific gravity being 0.969 at 53° Fahr. The best grades of the oil are colorless and vary from this standard to greenish-yellow in the lowest grades. At a temperature of 4° Fahr. (−18° C.) it becomes a transparent, yellow, solid mass. By exposure to the air it becomes rancid, thick, and at last dries up, forming a transparent varnish. It dissolves readily in its own volume of pure alcohol and is bleached by exposure to sunlight. Its use in soap-manufacture is chiefly for transparent soap.

Rosin.—Rosin in its various grades is the solid residue remaining from the distillation of crude turpentine, or resin, in which in its natural state it is dissolved. The oleo-resin is the sap of the pine-tree, the species of greatest economic importance in the United States being the *pinus palustris*. In regard to the function that the oleo-resin, which on removal from the tree forms crude turpentine, performs in the plant economy, it is essentially a waste product and plays no part in the processes of nutrition.

Rosin is an oxygenated hydrocarbon of indeterminate composition and acid properties. It is almost without smell and taste, lustrous, brittle, easily powdered, becomes soft at 176° Fahr., melts at 194°–212° Fahr., and is of the specific gravity 1.07. It varies in color from perfect transparency to black.

The naval stores industry comprises the recovery and sale of the products of the pine. The statistical year begins April 1st, and ends March 31st. The box-cutting season begins November 15th, and continues to March 15th, according to the law of Georgia, although the present tendency is to postpone box-cutting until the following month. The stilling-season begins early in March and continues to early December.

Box-cutting.—This is the first step in turpentine operating, and employs the turpentine labor during the winter. The box is a cavity 14 inches wide, 7 inches deep, and $3\frac{1}{2}$ inches from front to back, cut into the base of the tree by means of a long, narrow ax. This box has no other function than that of a receptacle for resin. Two, three, and sometimes four, boxes are cut in the larger trees. In the small trees the box is necessarily smaller, although larger relatively to the size of the tree.

Cornering.—Box-cutting is followed by cornering. This is done with an ordinary ax, a right-handed and a left-handed man working together. A slanting cut is made through the bark and about 1 inch into the sap-wood, the cut rising slightly from the top of the back of the box to a point perpendicularly above the corner of the box. By a side blow with the ax the wood is then split out between the cut and the rounding edge of the back of the box. The object of cornering is to provide a suitable surface for the subsequent scarification of the tree, and to direct the resin into the box.

Chipping.—In early spring the "chipping," or scarification of the trees, begins. It is continued weekly until November, and serves to open fresh resin ducts. The work is done by means of a "hack," a tool consisting of a flat steel blade in the form of the letter U. This blade, sharpened along its under edge, is fastened by means of a shank, at a right angle, into a wooden handle 1½ feet in length, on the end of which, to give greater momentum to the stroke of the "hack," is set an iron weight of from 5 to 7 pounds. The "chipper," standing squarely in front of the box, removes with the "hack" a strip of bark and sap-wood three-fourths of an inch wide just above the exposed surface produced by cornering, the laterally-inclined strokes being made from the right and from the left sides and penetrating the sap-wood about 1 inch at the deepest point. The freshly-exposed surfaces of sap-wood called the "streak," meet just above the centre of the box. The angular point thus formed is known as the "peak." The distance of the "streak" from the box increases with each weekly chipping. When the distance from the box is too great to admit of easy reach with the "hack," another tool, the "puller," is substituted, the result being the same in each case. Thirty-two streaks or chippings constitute a full season's work for the chipper.

Dipping.—Immediately after chipping the fresh resin appears and flows slowly into the box below. The flow is most rapid during the first two days after chipping; it then grows gradually less until after six or seven days no further flow takes place on most of the trees. At intervals of three or four weeks the resin in the boxes, called "dip," is transferred to large buckets by means of a flat metal instrument set on a long handle, and called a "dipper," or "dip" spoon. From the buckets the dip is emptied into barrels placed at suitable intervals. The filled barrels are then hauled to the distillery, or "still."

Scraping.—At the close of the chipping season the resin which has hardened on the exposed "face" of the tree, called "scrape," is removed by a sharpened flat tool and collected in barrels for distillation. This scrap contains approximately one-half as much spirits of turpentine as the dip from the boxes.

Raking.—After the trees have been scraped, each is "raked." This is a measure of protection against the annual burning of the grass, fallen pine-needles and undergrowth, which commonly occur throughout these forests. It is done by clearing of everything combustible a space of at least 3 feet around each tree, the tool used being, not a rake, but a broad hoe. This annual raking is a considerable item of expense to the operator; in spite of it, boxes are frequently burned out by the ground-fires.

The Crop.—The working unit in turpentine operating is the "crop," a tract of timber extending usually over an area of from 200 to 250 acres, and containing from 8,000 to 10,500 boxes, the number of boxes averaging two to the tree throughout the crop. The chipping of a crop constitutes the weekly task of one chipper. Usually the supervision of the work on from ten to fifteen crops is intrusted to one "woodsman."

Drifts.—For convenience the area of the crop is subdivided into "drifts," the limits of which are marked by lines blazed on the trees. The number of boxes in each drift varies between wide limits, though formerly 2,100 boxes constituted a drift.

Distillation.—One 25-barrel still suffices for the distillation of the products collected from twenty-five crops. The dip or scrape is placed in large copper stills, heated by direct flame from below. During distillation a stream of water flows into the still, from which the volatile spirits of turpentine are carried over, mixed with steam, into a large copper-worm, where they are condensed. The non-volatile rosin (colophony) remaining in the still is drawn from a tap at its lower edge into strainers, which remove chips, bark, sand, etc., the clear rosin passing into a vat below, from which it is dipped

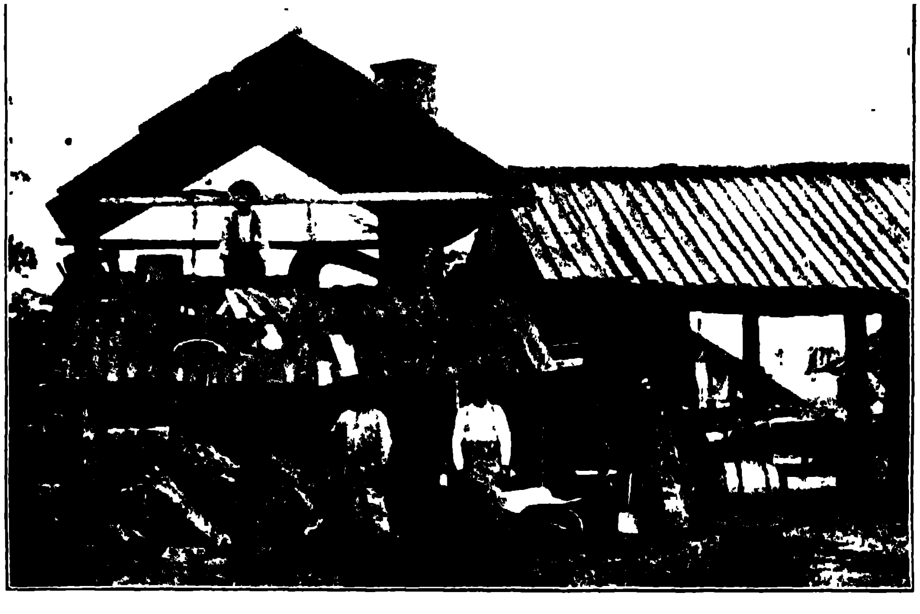

FIG. 20.—Rosin and Turpentine Still.

into barrels to cool and solidify. The rosin from first-year (virgin) dip is pale yellow; that from the later years of working is much darker. Pale rosins bring a much higher price than the darker or common rosins.

Turpentine gathering, as now conducted in the United States, is needlessly destructive of the forests and needlessly wasteful of the product. The method, under the box-system now universally employed, is to chop in the base of the tree itself a cup-like cavity, the sole purpose of which is to receive the resin which flows from a scarified face of the trunk above it. The box itself does not add to the flow of turpentine; on the contrary, experiment has proved that it diminishes the flow. It is an unnecessary wound driven into the body of the tree at its most vital spot, both weakening its vigor and lessening its power to support the strain of the wind. At the same time it opens the trunk to disease and provides a storehouse of combustibles against the coming of the forest's great enemy—fire. A forest which has been heavily turpentined by this method has before it only decay and death.

Rosin occurs in a number of grades with the quality usually indicated by letters as follows:

W. W.—Water White
W. G.—Window Glass
N.—Extra Pale
M.—Pale
K.—Low Pale
I.—Good No. 1
H.—No. 1.
F.—Good No. 2.
E.—No. 2.
D.—Good Strain.
C.—Strain.
B.—Common Strain.
A.—Black.

The grade of rosin is determined by the age of the "box" in which the crude turpentine is collected. W. W. and W. G. grades result from the distillation of the "virgin dip," or the first year's run; and with each subsequent year the crude turpentine collected from the same "box" becomes darker, less transparent and less fluid, until the fourth and last year it is very dark in color and yields on distillation the lowest grade of rosin.

A barrel of rosin varies greatly in gross weight, viz., from 250 to 600 pounds. On breaking up rosin at the factory, the staves, hoops and heads run from 15 to 20 per cent of the gross weight of the barrels. Quotations for rosins are for 280 pounds gross (no tares) and for rosins in lines as received by the factors, or brokers, in the naval stores yards on the river-front at Savannah. Factors do not select grades, but sell in the full lines as received by them. Buyers at the chief centres carry large stocks and frequently carry different grades of rosin several months before disposing of them. In the prices of rosins intended for export, interest, storage charges, insurance, handling, etc., enter. There is much dissatisfaction among all consumers of rosin, and spirits of turpentine as well, with the trade methods peculiar to the naval stores industry. This dissatisfaction among consumerr of rosin arises from:

(1) Unintelligible and incorrect marking of packages;

(2) False grading and fraudulent packing;

(3) Excessive weight of unnecessarily cumbersome containers;

(4) The arbitrary and senseless unit of weight (420 pounds, 400 pounds, 280 pounds, including package according to locality of origin).

The distinct branding of every package with the names of the producer and factor, together with clear weight-marks would fix the responsibility for fraud, error, or excessive weights.

The industry has been memorialized on different occasions by consumers of rosin in convention, to adopt a standard package with reasonably light staves and that the tare be branded thereon and to adopt a plan of billing rosin by the 100 pounds net.

A writer in an early number (about 1882) of the *Soap maker's Journal* which was later absorbed by the *Oil, Paint, and Drug Reporter*, presents the subject of rosin tares in the following words:

"As the consumer is expected to pay so much for each 280 pounds of rosin and wood, it is not only very essential that the wood part should be as light as possible, that is, that the packages should be made of thin staves bound by light-weight hoops; but it is important that the packages should be filled fairly to the top. In the case we speak of, many of the packages were only three-quarters full and a very large number were not over seven-eighths full. If the consumer paid the full rate per 280 pounds, he would be paying for a considerably larger amount of wood than was actually required for the packages.

He should not, in justice, be compelled to pay for any part of the staves, which could be sawed off above the heads without injury to the packages. The reader can more easily see the justice of the claim by supposing the case of a barrel of rosin which, when full, would weigh 350 pounds. It would contain say 300 pounds of rosin, and at $3 per barrel would make the rosin cost 1 cent per pound. Now suppose the same package to be only one-half filled. We would find the weight to be 200 pounds gross, or 150 pounds rosin, and at the same price per barrel the rosin would cost 2 cents per pound. Probably the most ignorant purchaser in the country would think he was not being fairly treated if he were to find he had purchased a lot of rosin in barrels only one-half full; but there are a great many very smart and intelligent soap-makers who never think of making a claim on rosin when the packages are but seven-eighths full, and yet the deduction for ullage should be very considerable, as we can readily prove by simple computation. Taking 350 pounds as the gross weight of a barrel when filled, or 300 pounds net of rosin, we find that if seven-eighths full there would be but 262½ pounds rosin net. Supposing the rosin to cost $3 per barrel we have the following result:

	Pounds, Gross.	Pounds, Net.	Cost Per Pound.
When full.	350	300	1 ct.
When ⅞ full	312½	262½	1.14 cts.
If only ¾ full.	275	225	1.33 cts.

"It may be that all of our readers have studied this subject, but we have our doubts."

Vegetable Soap-stock of Minor Importance.—As the use of vegetable oils in general in soap-manufacture is determined primarily by their parity with animal fats, we find in various countries different seed-oils used for this purpose. Palm, palm-kernel, sesamé, linseed, rape, and sunflower-seed oils, in addition to cocoanut-oil, are used extensively in European practice. With the exception of cocoanut-oil these oils are of limited use in the United States. These same remarks are also applicable to the recovered

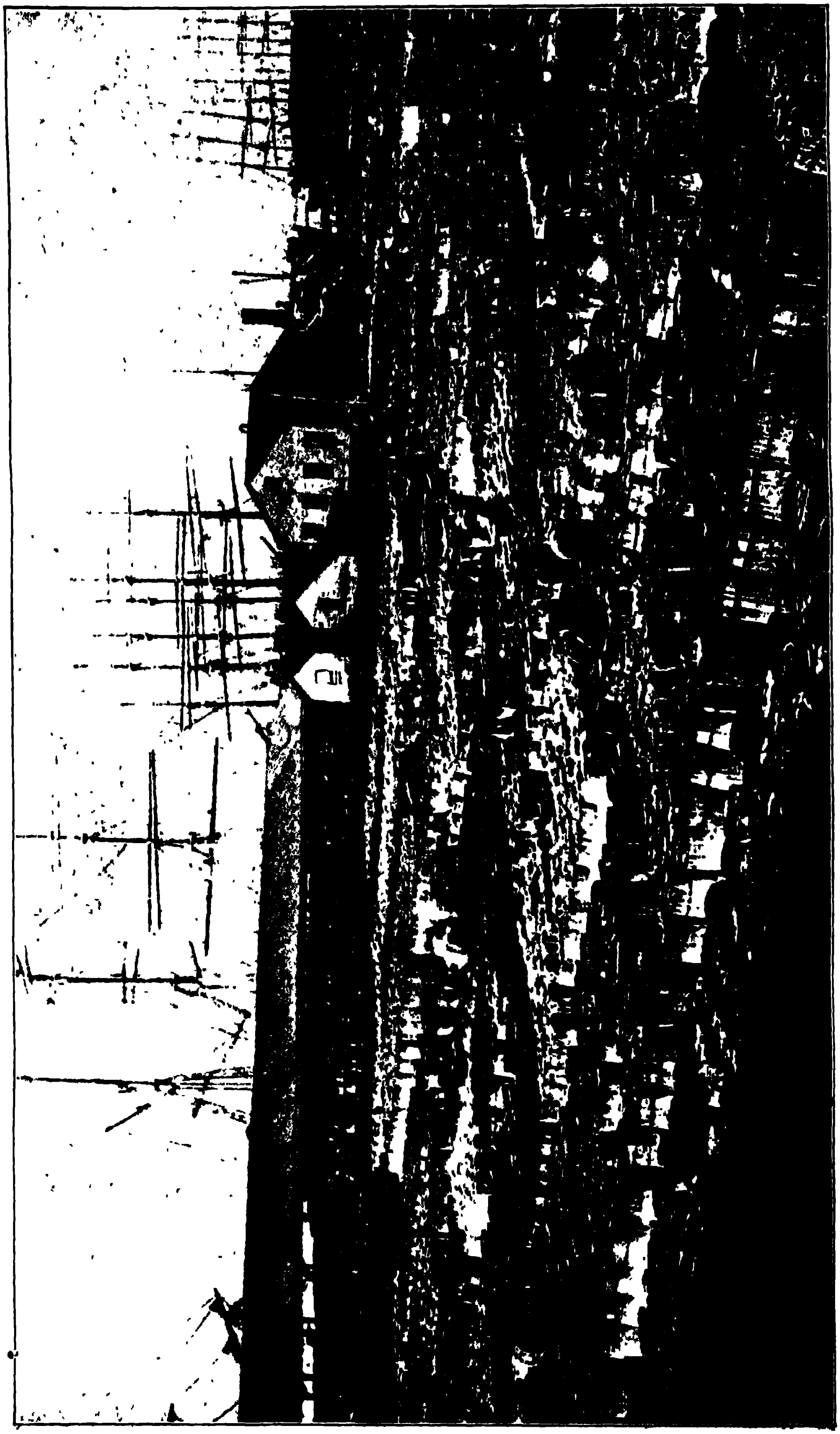

FIG. 21.—Naval Stores Wharf, Savannah.

greases used in soap and candle manufacture. Their recovery is determined by the relative commercial parity they may be able to hold with the bodies already available for these purposes.

The Alkali Industry.—By the "alkali industry" is meant the manufacture and sale of the products obtained directly from common salt. These comprise chiefly soda-ash, caustic soda, and bleaching-powder.

TABLE VIII.—OUTLINE OF THE LE BLANC PROCESS FOR THE MANUFACTURE OF SODA-ASH.

Stage.	Chemical Reaction.	Nature of the Process.
I.	$2NaCl + H_2SO_4 = Na_2SO_4 + 2HCl$	Common salt is treated with sulphuric acid with formation of Glauber's salt and hydrochloric-acid gas, which, absorbed by water in lead-lined towers, forms the muriatic acid of commerce.
II.	(*a*) $Na_2SO_4 + 2C = Na_2S + 2CO_2$ (*b*) $Na_2S + CaCO_3 = Na_2CO_3 + CaS$	Glauber's salt, coal and limestone are heated in suitable furnaces, whereby crude soda, or black-ash, is formed, with the liberation of carbon dioxide and formation of calcium sulphide, or lime waste, as a by-product.
III.	The black-ash is lixiviated with water and the sodium carbonate separated from solution by crystallization, with ten molecules of water, viz., $Na_2CO_3, 10H_2O$.	
IV.	The crystallized soda may come into market as soda crystals, or sal-soda, but is usually calcined to expel water, with the formation of the anhydrous sodium carbonate or soda-ash.	

The growth of the soap industry is parallel with the development of the alkali industry, for the latter means the production of a cheap and pure saponifying agent. With the exception of the product obtained by the lixiviation of very impure natural deposits of the alkaline carbonate, previous to 1823, the sole commercial source of the carbonates of potash and soda was the seaweeds, barilla, and kelp. In medical alchemy, "kali," "natro," and "soda" denoted the same thing, viz., fixed alkali of whatever origin. Stahl (1702) first recognized the distinction between alkali and natron, or what we now term respectively alkali and common salt. Duhamel (1736) established the distinction between the so-called vegetable and mineral alkalis. As a result of the embargoes of the French Revolution, France was cut off from the supply of kelp and in 1775 the French Academy of Sciences offered a prize for a successful process for converting salt into alkali. Among the many methods De la Metherie, about 1787, proposed to calcine sodium sulphate with charcoal. This, however, gave little more than sulphide. In 1790 Le Blanc supplied limestone and received the merits of the invention. In 1791 the French Academy granted to Le Blanc a patent for fifteen years, but business reverses fell upon him and he never lived to enjoy the results of his ingenuity. In 1806 at the age of fifty-three he committed suicide in a mad-house, the inventor of one of the most important chemical processes in the history of the world.

After the repeal of the salt duties the manufacture (on a large scale) of soda-ash by the Le Blanc process was begun in England by James Muspratt in 1823, but it was not until 1854 that William Gossage began the commercial production of solid caustic soda. The use of the now common sheet-iron drums was introduced by Thompson in 1857. With the recovery of the by-products, the Le Blanc process is complicated and possesses many sanitary and technical disadvantages.

The "carbonating tower" of the ammonia or Solvay process, shown in section in Fig. 22, is built of cast-iron rings or segments, *AA*, 3.5 feet high and 6 feet in diameter, to a height of 50 to 65 feet. In the bottom plate of each segment is a hole above which is a perforated dome-shaped diaphragm. Each segment is provided with a cooling-water pipe to counteract the heat of chemical action. Ammoniacal brine under pressure is forced through the pipe *P*; cooled carbon dioxide is forced through the pipe *C* in the bottom segment. By means of the perforated diaphragms it is brought into intimate contact with the ammoniacal brine. The precipitated sodium bicarbonate accumulates in the bottom of the tower where it is drawn off as a thick, milky liquid at *H*. This product is evaporated to dryness and calcined as indicated in the accompanying outline, II, Table IX.

The production of electrolytic caustic soda is based upon the principle that when a current of electricity is passed through a solution of common salt, the latter is decomposed into its constituent elements, sodium and chlorine. The success of the various electrolytic processes that have been developed in the past few years depends upon the nature and efficiency of the diaphragm used to separate these two elements and the products formed from them. In the Castner-Kellner process, which is the most promising and is the one most extensively used in this country at the present time, no diaphragm is used and mercury is employed as a movable cathode.

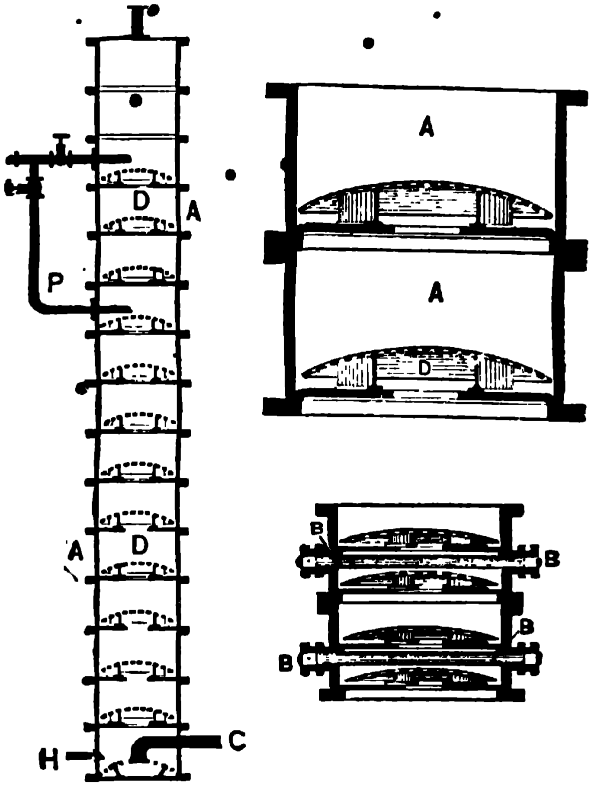

FIG. 22.—Carbonating Tower. Solvay Process.

On an iron plate (Fig. 23), supported at one end on a knife edge, and to which, by means of an eccentric, *E*, a slight rocking motion may be given, is built a closed-in rectangular vessel of slate 3·5×6 feet, divided into three compartments. These compartments communicate with each other by means of V-shaped grooves formed at the bottom of

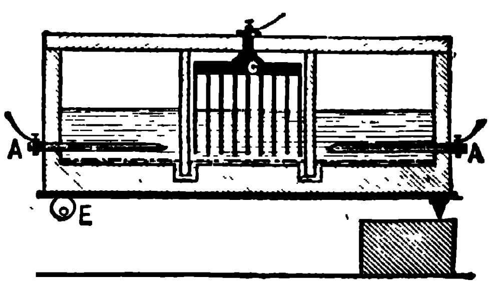

FIG. 23.—Castner-Kellner Electrolytic Cell.

the dividing walls; the floor of these troughs is the iron plate previously mentioned. The outer compartments suffer the most motion during the rocking, and each in itself constitutes a cell for the electrolysis of salt solution. The anodes, *AA*, are of carbon, and they are supported in the centre of each of the outer cells; the cathode is of mercury, resting on the floor of the vessel, and just sufficient rocking is given to the apparatus to enable this mercury to form, at the one time, a thin layer on the floor of the central compartment and also on the floor of the other side cells. The mercury in the connecting grooves forms lutes which effectually prevent liquids in any one division escaping into adjoining portions.

Supported above the mercury in the central compartment is an iron wire gauze, *C*, connected electrically with the iron plate on which the whole rests.

To decompose salt by this process the two outer cells are filled with concentrated brine, and the middle one with a 20 per cent caustic soda solution, which is chosen, since at this strength the conducting power of caustic soda solution reaches a maximum. Passage of the current liberates chlorine at the anode of one outer cell, and, at the cathode, causes the formation of a corresponding amount of sodium amalgam. Rocking the apparatus causes this amalgam to flow into the central compartment, in which itself becomes the anode, the contained sodium being partially oxidized to give caustic soda, while hydrogen is evolved from the iron gauze cathode. The amalgam deprived of its sodium flows, as the rocking is continued, into one or other of the outer cells, where a further electrolysis of the salt solution occurs, and the amalgam formed is again decomposed as above.

For constant working the brine in the outer cells must be kept at constant strength, and well circulated to prevent rise of temperature; sufficient water must be admitted into the central decomposing cell to allow of a constant flow from this of a 20 per cent caustic solution.

The resistance of the cell is low, and the efficiency high, about 80 per cent of the current being converted into chemical energy, which is used up in the cell; the electromotive force of polarization is, however, high. The caustic soda solution obtained is of a very high degree of purity, and is prepared directly of a strength of 24°

Bé.—this is much higher than that of the causticized liquor of the Le Blanc process.

The main point to be observed is the employment of a very pure brine; the presence of magnesium or calcium salts causes these metals to be separated from the solution, and to amalgamate with the mercury. With the accummulation of these the mercury loses its power of running freely; as the apparatus rocks patches are left on the slate bottom, and are rapidly attacked by the chlorine present in the anode-containing compartments. It is therefore necessary to keep the mercury clean, and it has been found advisable on the manufacturing scale to thoroughly clean each cell periodically.

TABLE IX.—OUTLINE OF THE AMMONIA PROCESS FOR THE MANUFACTURE OF SODA-ASH.

Stage.	Chemical Reaction.	Nature of the Process.
I.	$NaCl + NH_4OH + CO_2 = NaHCO_3 + NH_4Cl$	Ammoniacal solution of salt is saturated with carbon dioxide in carbonating towers, Fig. 22. Products are sodium bicarbonate and the by-product ammonium chloride.
II.	$2NaHCO_3 + (heat) = Na_2CO_3 + H_2O + CO_2$	The bicarbonate of soda is converted into the normal sodium carbonate by calcination with liberation of water and carbon dioxide.
III.	$NH_4Cl + CaO \cdot H_2O = 2NH_4OH + CaCl_2$	Ammonium chloride, by-product of the first stage, is deprived of its ammonia by treatment with lime, with formation of the waste product, calcium chloride.

In 1863, the mechanical difficulties surrounding the production of soda-ash by the ammonia, or Solvay process, were overcome by Ernest and Alfred Solvay, who at that time established works for its operation in Belgium. Since then works have been established all over the world and at the present time the greater part of the soda-ash consumed is made by this process.

The principle of the process is the precipitation of the sodium

TABLE X.—COMPARISON OF ALKALI MANUFACTURING PROCESSES.

DISADVANTAGES.

Le Blanc	Ammonia, or Solvay.	Electrolytic.
General complication of process and troublesome recovery of by-products, which have been of comparative recent introduction, made imperative by competition. Necessity of associated sulphuric acid manufacture. Large expenditure of manual labor and heat. Presence of impurities absent in the ammonia-soda.	Total loss of chlorine contained in the salt; no completely satisfactory process has as yet been devised to obtain this from the calcium-chloride waste. Unsuitability of sodium bicarbonate to be converted into caustic soda by the lime process; this may, however, be overcome by the adoption of the Lowig "Ferrite" process, according to which solid alkaline bicarbonates, in contact with oxide of iron, are decomposed at a bright red heat with evolution of carbonic anhydride and formation of an alkaline ferrite. This ferrite is insoluble in cold water, but on boiling it is gradually resolved into caustic alkali and oxide of iron, which is filtered off and re-used in the process.	Comparatively high cost of installation; as only a very moderate current density can be used at the electrodes of a cell in which a salt solution is being decomposed, it is necessary to have a large number of cells for even a moderate output, with the result that cost of plant per ton of material produced per day is high. High rate of depreciation. The caustic soda and chlorine do not separate neatly and completely from the menstruum in which they have been formed, in many of the proposed cells; many secondary bodies are formed. This is not the case, however, with the Castner-Kellner
Repairs are of a heavy character		
ADVANTAGES.		
Production of hydrochloric acid and of chlorine therefrom for the manufacture of bleaching-powder.	Purity of product. Absence of offensive by-products and conditions of manufacture. Less labor expenditure.	Only common salt and water are necessary for the production of caustic soda and chlorine, hence there is no waste product and no necessity of intermediate processes for the liberation of chlorine from by-products and for the formation of caustic soda by causticization from soda-ash. Minimum expenditure of labor and heat. Purity of products. Cleanliness of operation.
Large output with comparatively few units of plant of large size and of comparatively simple construction. Repairs are not numerous nor applied to a great number of small units.		

GENERAL CONSIDERATIONS.

The present consumption of alkali amounts to six times that of bleaching-powder. The formation of these two products in theoretical quantity from salt would yield two tons of bleach for every one of alkali. It is thus very evident that, to satisfy the world's demand for alkali, the manufacturer must be in such a position that he can afford to throw away the greater part of his chlorine, if he cannot do this, he must limit his output of alkali to the demand for bleach.

The Le Blanc process depends on the chlorine for a large share of its profit; all electrical processes must do the same. The ammonia-soda process alone can disregard this element, and is, therefore, par excellence, the soda-making process of the future, and is safe from all competition, and such competition can only rest between the Le Blanc and the various electrical methods.

acid carbonate in an ammoniacal solution of common salt by means of carbonic acid gas. The salt is obtained in solution from driven wells; the ammonia is a coke-oven by-product; the carbonic acid gas is obtained in the manufacture of lime which is itself used to set free the ammonia obtained as a chloride by-product in the process. The various materials are employed in aqueous solution or suspension and the plants are adapted for the handling of immense quantities of liquids.

Caustic Soda.—In the Le Blanc process, caustic soda is manufactured from the black-ash liquor, obtained in stage III, as indicated in the outline, Table VIII, of the process, by causticization with lime. The density of the liquor must not exceed 13° Bé., or the reaction will be reversed by the calcium carbonate being attacked by the caustic soda. The reaction of causticization is expressed by the following equation:

$$\underset{(106)}{Na_2CO_3} + \underset{(74)}{(CaO.H_2O)} - \underset{(100)}{CaCo_3} + \underset{(80)}{2NaOH}.$$

This impure solution of sodium carbonate is heated to boiling and the lime added, with agitation by means of an air-blast. When causticization is complete, the contents of the vats are allowed to settle and the supernatant liquor is drawn off from the lime mud and evaporated to dryness and to fusion, the impurities having been largely removed in the meantime by oxidation and fractional crystallization. Nitre is used to oxidize the thiosulphate to normal sulphate; zinc oxide may be used to precipitate sulphides in accordance with the following reaction:

$$Na_2S + ZnO + H_2O = 2NaOH + ZnS.$$

A sample representing the contents of the kettle is tested for total alkali. If found to be below 60 per cent quality the contents are worked up to that test. If the test indicates the quality to above 70 per cent the product may be worked into the highest grade made, or into that grade testing most closely to the contents of the kettle. The reduction of quality to the grade of caustic desired is effected by the addition of salt.

The molten caustic is run directly into sheet-iron drums. Causticization of soda-ash with lime is most complete in dilute solutions, as shown by the following results of experiments carried out by Prof. Lunge:

Per Cent Na_2CO_3 in Liquor.	Sp. G. Before Causticizing.	Per Cent Na_2CO_3 made Caustic by Treatment	
		I.	II.
2	1.022 at 15° C.	99.4	99.3
5	1.052 " "	99.0	99.2
10	1.107 " "	97.2	97.4
12	1.127 " "	96.8	96.2
14	1.150 " "	94.5	95.4
16	1.169 " 30° C.	93.7	94.0
20	1.215 " "	90.7	91.0

Sixty pounds of pure quick-lime is sufficient to render caustic 100 pounds of 58 per cent soda, but as lime varies very much in quality, and is commonly not pure, more than sixty pounds is usually required. The proper way is to test the liquor for causticity before the boiling with lime is completed. To do this, some of the liquor should be taken out and allowed to settle. Then take a little of the clear liquor in a test tube and drop into it a few drops of dilute sulphuric acid. If bubbles of gas are liberated, it shows that more lime is required, and the boiling should be continued until the clear liquor gives no bubbles on the addition of sulphuric acid.

Causticization of Soda-ash by the Soap-manufacturer.—The manufacture of dilute caustic soda solution, upwards of 14° Bé., by the soap-manufacturer himself is determined by the quantity of caustic soda consumed, the amount of the disparity in price between the caustic soda in solution reduced to the required density—and the solid caustic soda in drums, and his appreciation of the convenience and cleanliness of use of the latter form.

The manufacture and concentration of dilute caustic-soda solution by the Jobbins process are shown in the following diagrams, and descriptions.

Commercial Grading of Soda-ash and Caustic Soda.—The system of grading alkali and caustic is based upon the molecular composition of these bodies, and the quotations of the various grades in terms respectively of 48 per cent. alkali and 60 per cent. caustic is a vestigial characteristic of the early Leblanc days and an evidence of the highest grades of those products they were then mechanically able to produce. The molecular weight of sodium carbonate, Na_2CO_3, is 106, composed of 62 parts by weight, or 58.49 per cent. of Na_2O, the remainder being CO_2. A soda-ash that contains 58.49 per cent. of Na_2O is therefore chemically pure, this percentage being equivalent to 100 per cent. Na_2CO_3. A 58 per cent. alkali should contain 58 per cent. of Na_2O or its equivalent, 99.16 per cent. Na_2CO_3; likewise a 48 per cent. alkali should contain 48 per cent. Na_2O or its equivalent, 82 per cent. Na_2CO_3. The reduction of any grade of soda to that of 48 per cent. is effected by admixture with common salt. There are present for comparison two fairly representative analyses of these two standard grades of soda-ash:

Grade.	Per Cent. Na_2CO_3.	Per Cent. NaCl.	Per Cent. Na_2SO_4.
48%	60.64	28.34	4.35
58%	98.72	.54	.20

Grade.	Per Cent. NaOH.	$F_2O_3Al_2O_3SiO_2$.	$CaCO_3MgCO_3$.	H_2O.
48%	1.29	1.12	Traces.	4.26
58%	....	.10	.17	.26

Caustic soda occurs on the market in a variety of grades and is sold on the basis of 60 per cent. of Na_2O. Caustic soda as a product of the alkali industry did not arise until thirty years after the industry was established in Great Britain, and the expression of its quality in the same terms as that of soda-ash might be expected. The molecular weight of caustic soda is 40; to arrive at sodium oxide, Na_2O, as an expression of the customary unit two molecules with a total molecular weight of 80 are used. In $2NaOH$ there are 62 parts, or 77½ per cent. Na_2O, the remainder being H_2O. Therefore a chemically pure caustic soda contains 77½ per cent. Na_2O, or its equivalent, 100 per cent. NaOH. There is for purpose of comparison in the following table the percentage of the essential ingredient corresponding to, but never present in, the various grades of caustic commonly found in the market:

Grade.	Per Cent. NaOH.
60-degree	77.42
70 "	90.32
72 "	92.90
74 "	95.48
76 "	98.06
77 "	99.35
77½ "	100.00

Sodium chloride, sodium carbonate, and sodium sulphate, in varying proportions, constitute chiefly the remainder of the ingredients. With the present system of grading based upon the chemical determination of the total alkali, the Na_2O of the Na_2CO_3 is estimated with the Na_2O, in terms of which the caustic soda or NaOH is expressed. With this method of expressing the quality of the caustic, the soap-maker has just cause for complaint in that a variable percentage of a worthless ingredient is included in the total percentage of the essential ingredient present. The following is an analysis of a sample of commercial caustic purporting to be of 74 per cent. quality:

Total alkali estimated as Na_2O	74.18%	
Caustic alkali " " "	69.88%	
Caustic alkali " " NaOH		90.18%
Combined alkali " " Na_2CO_3		7.35%
Combined alkali " " Na_2O	4.30%	
Sodium chloride " " NaCl		2.02%

This analysis indicates sample to be of substantially 70 per cent. quality. The difference between the sodium hydrate actually present and that claimed, viz., 4.30 per cent. Na_2O, is due to the 7.35 per cent. Na_2CO_3, this being estimated as its equivalent, 4.30 per cent. Na_2O, in the total sodium oxide. As more or less carbonate is invariably present in all commercial caustic, especially in the lower grades, the system of including it in the expression of the quality of this product is open to severe criticism. Quotations of quality are thus confessedly a misrepresentation. The only rational method is the expression of the Na_2O as free caustic, or preferably units of NaOH.

This would be an absolute index of the value of the caustic as a saponifying agent, and not, as by the method in vogue, an uncertain approximation of the same. English degrees indicate the strength of the ash or caustic in terms of Na_2O, but, owing to either a wilful or accidental error in atomic weights, English analyses indicate a greater percentage of Na_2O than is actually present. This error has become so firmly established by tradition that modern ideas have been unable as yet to eliminate it. In Germany and Russia the strength is expressed in terms of sodium carbonate. This system is perfectly rational when applied to soda-ash, but is inconsistent when applied to caustic. The expression of the value of commercial caustic soda in terms of an impurity, which, in so far as the soap industry is concerned, is positively worthless as a saponifying agent for neutral glycerides, is certainly not conducive to clear ideas on the subject.

The superior advantages and economy of high-grade caustic need no argument. It is true of this product that the best within certain limits is the cheapest. There is presented in the following table the price per pound of sodium hydrate as it occurs in the customary grades of caustic, assuming that no carbonate is estimated as caustic:

CAUSTIC SODA.

Grade.	Price per 100 Pounds.	Per Cent. NaOH Present.	Price NaOH per 100 Pounds.
60%..................	$1.65	77.42	$2.131
70%..................	1.50 for 60%	90.32	1.937
74%..................	1.60 for 60%	95.48	2.066
76%..................	1.70 for 60%	98.06	2.196

The anomalous variation in prices quoted arises from the slight differences in cost of production of the lower grades, combined, for those grades, with the proportionally greater cost of packages, transportation, etc.

The total charges contingent upon marketing a 60 per cent. caustic are the same as those of a 70 per cent., although the former contains considerably less of the essential ingredient; also the cost of production of a 70 per cent. caustic is but little more than that of 60 per cent. The increased cost of production of the higher grades, viz., 74 per cent. and 76 per cent., makes necessary a higher price, which is less than it would be if cost of marketing were correspondingly increased.

The system of quoting the higher grades in terms of 60 per cent. caustic is a peculiarity of the trade, and the fractional increase in price based upon the degree is a measure of the increased cost of carrying the manufacture of the product above the 60 per cent. grade.

In the preparation of caustic lyes of different densities from various grades of caustic, the influence of the impurities, chiefly sodium chloride, sodium carbonate, and sodium sulphate, is to reduce the active value of the solution for the specific gravity indicated. This reduction in saponifying power is least for the highest grades and greatest for the lowest as a natural result of the increased percentage of these impurities present. The following table indicates the percentage of sodium hydrate present in lyes of different densities, made of the usual grades of caustic, corresponding to the densities of lye made from chemically pure caustic. It is assumed that the total alkali is present entirely as caustic, which never actually occurs. The figures stated, for reasons previously given, are generally slightly higher than would be found in practice. However, the table possesses value as a basis of comparison, and for all technical purposes the figures are sufficiently accurate.

Causticization of Soda-ash.—The consumption of caustic soda for oil-refining and soap-making may be of sufficient amount, together with other economic considerations, to warrant the manufacturer undertaking the manufacture of caustic soda himself. Caustic soda in aqueous solution is obtained from soda-ash, i.e.,

TABLE XI.—PERCENTAGE OF SODIUM HYDRATE IN LYES MADE FROM VARIOUS GRADES OF COMMERCIAL CAUSTIC.

Specific Gravity.	Degrees Bé.	Grades of Caustic.						Degrees Twaddell.
		77½°	76°.	74°.	72°.	70°.	60°.	
		Per Cent. NaOH.						
1.075	10	6.55	6.42	6.25	6.08	5.91	5.06	15.0
1.083	11	7.31	7.17	6.98	6.79	6.60	5.66	16.6
1.091	12	8.00	7.84	7.63	7.43	7.22	6.19	18.2
1.100	13	8.68	8.51	8.29	8.06	7.84	6.72	20.0
1.108	14	9.42	9.24	8.99	8.75	8.51	7.29	21.6
1.116	15	10.06	9.86	9.60	9.34	9.08	7.78	23.2
1.125	16	10.97	10.76	10.47	10.20	9.91	8.49	25.0
1.134	17	11.84	11.61	11.31	11.00	10.69	9.17	26.8
1.142	18	12.64	12.40	12.07	11.74	11.41	9.78	28.4
1.152	19	13.55	13.28	12.93	12.59	12.24	10.49	30.4
1.162	20	14.37	14.09	13.72	13.35	12.97	11.12	32.4
1.171	21	15.13	14.84	14.44	14.06	13.67	11.71	34.2
1.180	22	15.91	15.61	15.19	14.78	14.30	12.31	36.0
1.190	23	16.77	16.44	16.01	15.58	15.15	12.98	38.0
1.200	24	17.67	17.33	16.87	16.42	15.96	13.68	40.0
1.210	25	18.58	18.23	17.74	17.27	16.78	14.38	42.0
1 220	26	19.58	19.20	18.69	18.19	17.68	15.16	44.0
1.231	27	20.59	20.19	19.66	19.13	18.60	15.94	46.2
1.241	28	21.42	20.99	20.44	19.89	19.33	16.57	48.2
1.252	29	22.64	22.20	21.62	21.03	20.45	17.53	50.4
1.263	30	23.67	23.21	22.60	21 99	21.37	18.32	52.6
1.274	31	24.81	24.33	23.69	23.05	22.42	19.21	54.8
1.285	32	25.80	25.30	24.63	23.96	23.30	19.97	57.0
1.297	33	26.83	26.31	25.62	24.92	24.23	20.77	59.4
1.308	34	27.80	27.26	26.55	25.82	25.11	21.52	61.6
1.320	35	28.83	28.28	27.53	26.79	26.04	22.31	64.0
1.332	36	29.93	29.35	28.58	27.81	27.04	23.17	66.4
1.345	37	31.22	30.62	29.82	29.00	28.46	24.40	69.0
1.357	38	32.47	31.84	30.99	30.16	29.32	25.13	71.4

commercial sodium carbonate, by treating the latter, under suitable conditions, with slaked lime. The procedure of manufacture is in accordance with the following chemical reaction:

$$\underset{106}{\mathrm{Na_2CO_3}}\text{(soda-ash)} + \mathrm{Ca(OH)_2}\text{(slaked lime)}$$

$$= \underset{100}{\mathrm{CaCO_3}}\text{(lime-mud)} + \underset{80}{\mathrm{2NaOH}}\text{(caustic soda)}.$$

Thus, theoretically, 106 parts of sodium carbonate treated with 74 parts of calcium hydrate yield 100 parts of calcium carbonate and 80 parts of sodium hydrate. On a practical scale the theoretical yield is only approximately attained.

Description of Plant.—In Fig. 25 is shown, in plan and elevation, the mechanical equipment of a causticizing plant, consisting of the converting-kettle, screen-tank, lye storage-tank, evaporator, pumps, etc. Fig. 24 is a conventional form of lime-house. Fig. 27 is an enlarged sectional view of the screen-tank; and Fig. 26 is an enlarged sectional view of the lower portion of the converting-kettle.

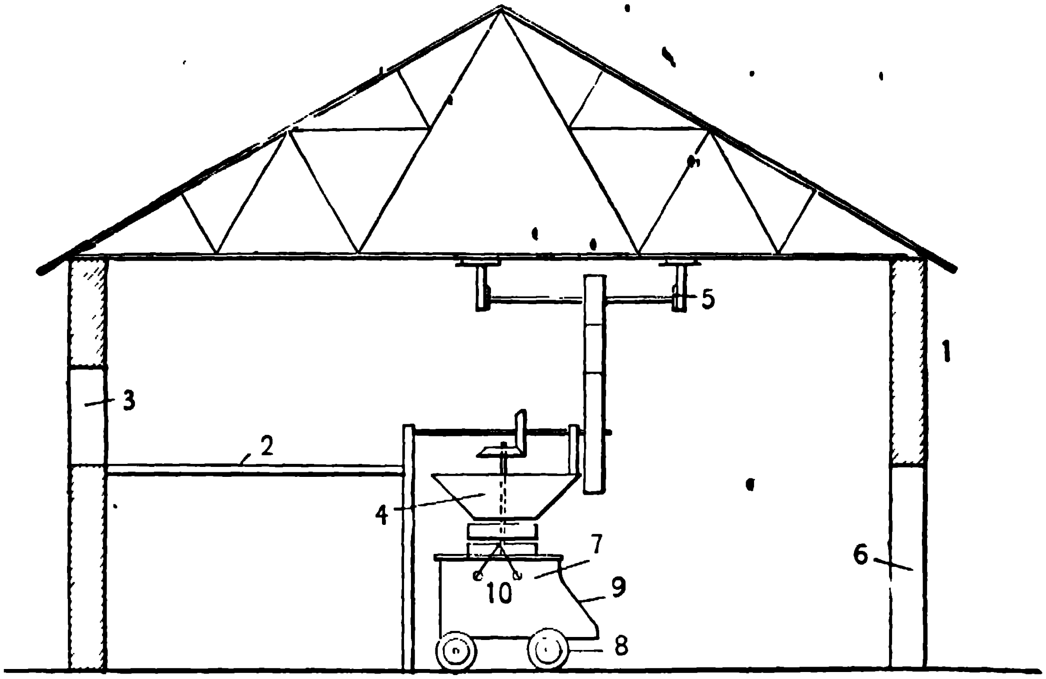

FIG. 24.—Conventional Form of Lime-house.

It is best to provide a building or a suitable fire-proof room or inclosure for the storage and preparation of the lime to be used in the converting-kettles, and as imperfectly burnt or inferior qualities of lime often contain stone or unburnt material, such is preferably crushed before being passed to the converting-kettle, as it also affords an easier, more rapid, and complete operation in the converting-kettle.

In Fig. 24, 1 represents the lime-house, having a raised or elevated iron floor 2 for the storage of the lime. Adjacent to one side of this elevated floor is a door 3, through which the lime can be received.

4 is a lime-mill or crusher operated by a suitable power, a counter-shaft 5 being shown. The crushed lime from the mill may be passed directly into the carrier 7, which may be of perforated iron if it is to be used as a basket inside of the converting-kettles,

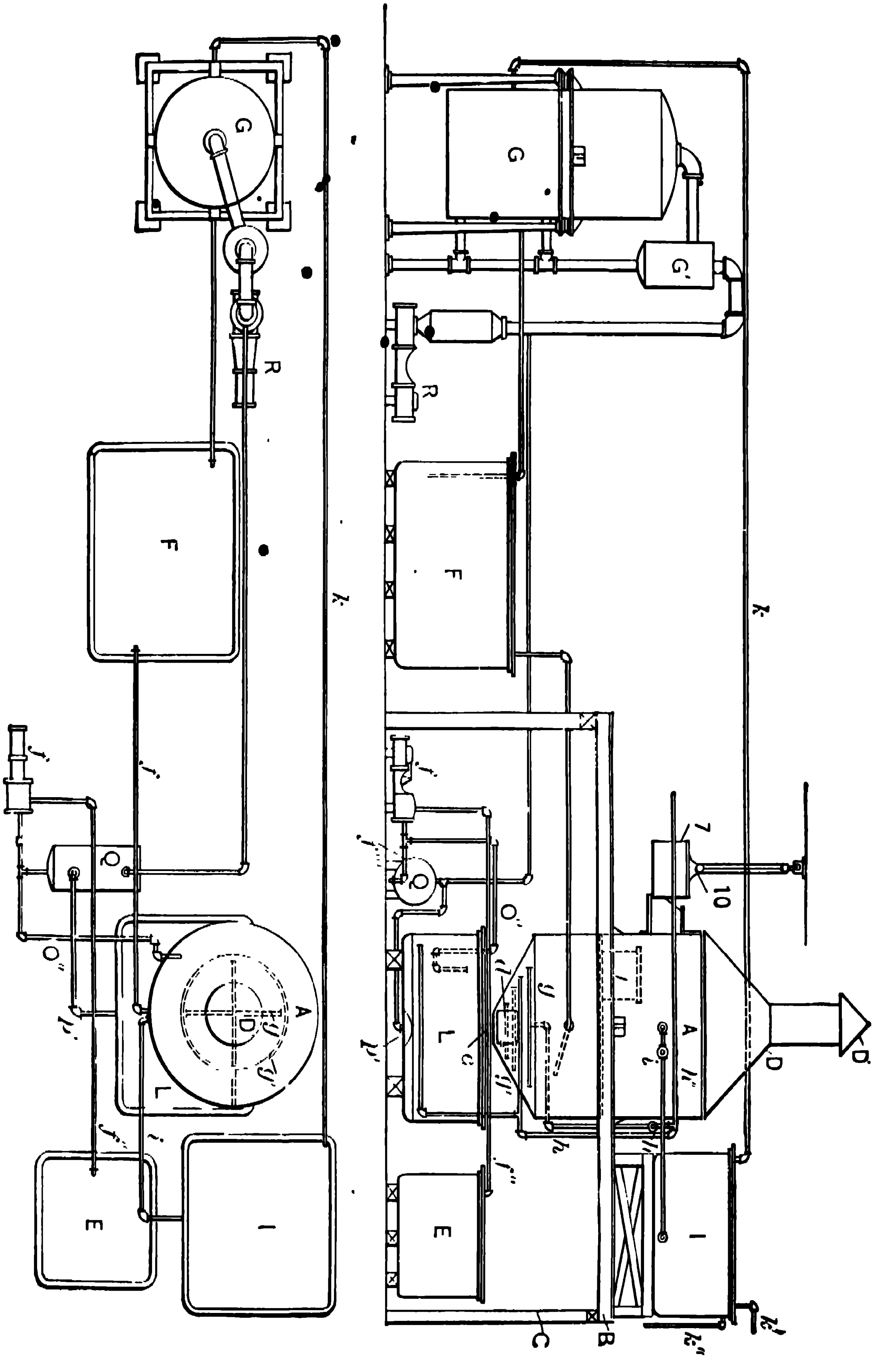

Fig. 25.—Diagram of Apparatus for the Causticization of Soda-ash and Concentration of Caustic Liquor.

or the carrier may be provided with double-flap doors 9 when it is to be used as a chute.

8 is a wheeled frame for carrying the basket or chute to the converting-kettle. In lieu of this wheeled frame an overhead tramway may be used, as is shown in Fig. 25.

The carrier 7 is provided with suitable swivel-handles 10, by means of which the carrier can be hoisted and its contents emptied.

6 is the entrance from the lime-house to the converting-kettle.

The lime-carrier 7 is preferably made of iron or a non-inflammable material, and as the floor in front of the iron converting-kettle is bricked or covered with iron or otherwise made fireproof no danger is incurred by using this carrier in an ordinary building, thus enabling this part of the operation to be carried on in an ordinary factory building.

By the use of power the lime can be conveyed to the converting-kettle by means of any suitable endless conveyor or to any conveniently located receiver, from which it can be either by hand or automatically fed into said converting-kettles.

In Figs. 25 and 26, *A* is a converting-kettle formed of any suitable shape and with a conical, bevelled, or flat bottom, as may be desired. This kettle is supported by means of lugs from the I-beam frames *B*.

C are suitable iron pillars for supporting the beams *B*.

The top of the kettle is preferably covered with a hood *D*, formed of wood or metal, at the top of which is an exit *D′* for the passage of the steam from the kettle. Near the bottom of the converting-kettle is shown an iron door *d* for the removal of the residuum. This door when used is preferably shaped to conform to the kettle and is carried down as nearly to the bottom of the kettle as is possible. At the extreme bottom of the kettle is a valved outlet-pipe *e*, also for the removal of the residue.

f is a pipe having a pivoted elbow within the kettle, by means of which the liquid lye can be run into the receiving-tank *F*.

Located at the bottom of the kettle *A* is preferably a perforated coil of steam-pipe *g*, through which steam can be ejected for the boiling of the caustic lye, and it can also be used for producing motion of the contents of the kettle. In addition to this a closed coil of steam-pipe *g′* may be employed for heating the contents of the converting-kettle. If the perforations in the steam-pipe *g* are placed underneath the pipe and on the sides thereof at about an angle of 45°, there will be a thorough agitation of the material, obtaining a complete conversion. The pipes *g* and *g′* are located at any suitable distance above the bottom of the kettle.

Air under pressure may be injected into the liquid; or to attain the same end a mechanical mixer or stirrer can be introduced within the kettle, the object being to maintain a constant agitation of the contents of the kettle and the complete admixture thereof. The pipes *g* and *g′* are connected with a source of steam-supply by means of the pipe *h*, having the valve *h′* therein. *h″* is the main steam-pipe, shown running across the front of the kettle. If more than one kettle is used, this pipe can be extended for the supply of steam to the additional kettles.

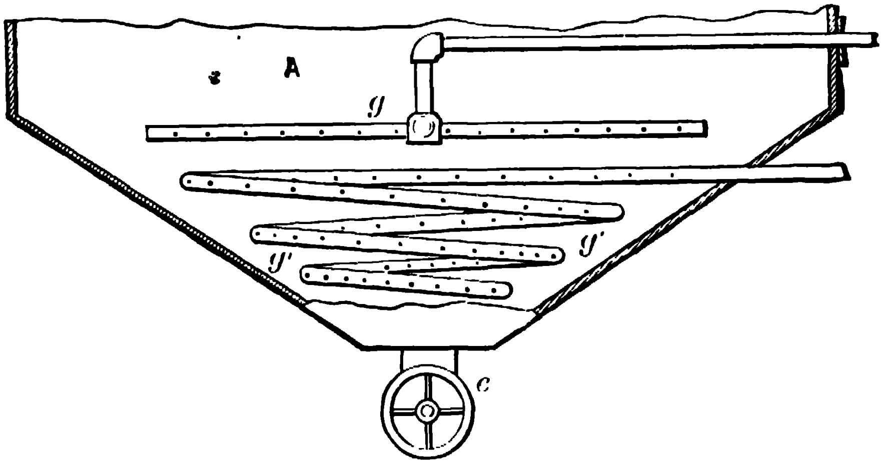

FIG. 26.—Sectional View of Lower Portion of Converting-kettle.

Below the hood *D* is an opening, preferably in front, for the introduction of the ash and lime. The latter is preferably introduced in small quantities at a time into the basket *j*, which is adjacent to said opening, so located as to be partially immersed within the liquid. The entrance to the kettle is protected by a door, which being closed when not in use will prevent the outlet of the steam. As shown, the hood *D* should extend a short distance beyond the sides and over the top of the kettle, so that the draft thereby produced will facilitate the exit of the steam through the opening *D′*. The hot-water receiving-tank *I* is elevated, so that its contents can be run into the converting-kettle *A* by means of the valved pipe *i*.

This tank is supplied with hot water from the coils of the evaporator or concentrator *G* by means of the pipe *k*. If this supply is insufficient, additional water, either hot or cold, may be introduced by means of the valved pipe *k'*.

k'' is an overflow from the tank *I*.

Situated below the converting-kettle is the screen-tank *L*, into which the residue of the materials used can be emptied by means of the pipe *c* or the door *d*. This tank is preferably provided with a false suspended bottom *o* (see Fig. 27) consisting of one or more layers of canvas and wire-gauze and which is so arranged that the residue or lime-mud will not pass through the screen when operated upon by a vacuum-pump, the liquid and air passing through a pipe connecting with the bottom of the tank. This false bottom is preferably placed two or three inches above the bottom of the tank and consists, first, of a wire screen *o'* of very fine mesh, underneath which the duck or canvas *o''* is stretched. The fabric is supported, preferably, upon a sheet of gauze *o'''* of slightly coarser mesh, which in turn is supported upon an iron frame *o''''*. Within this tank, a short distance above the false bottom, is a perforated steam-pipe *O'*, the perforations of the pipe being preferably arranged underneath and on the sides thereof at an angle of about 45°.

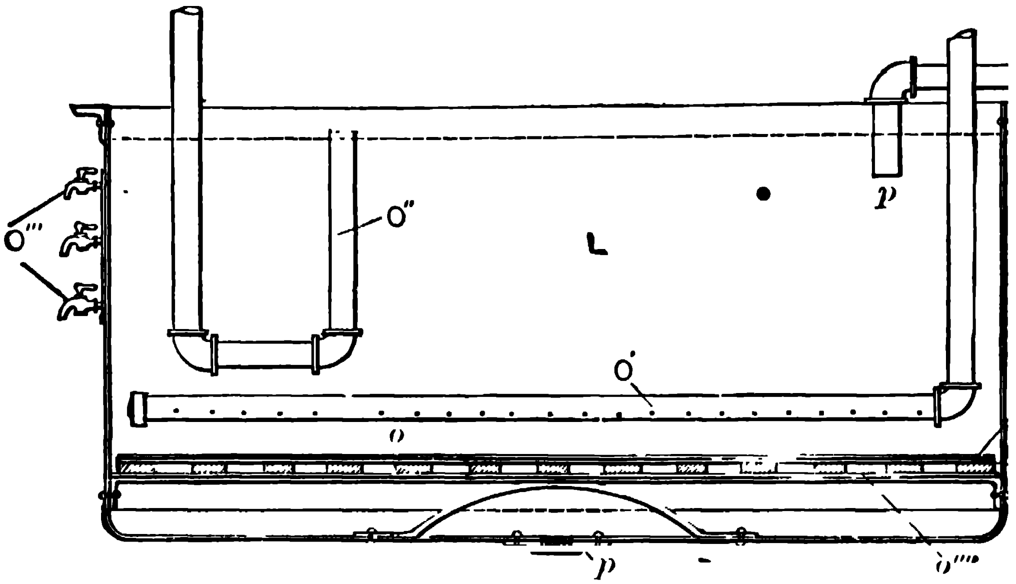

FIG. 27.—Sectional View of Screen-tank.

A suitable jointed elbow *O''* is used for decanting such portion of the supernatant liquid and wash-water as settles above the residue.

O''' are faucets or cocks used for accomplishing the same purpose. A pipe *p* for the supply of water for the admixture and the washing from the lime-mud or residue of any caustic lye not removed in the kettle is placed at one side of the tank. Adjacent to the screen-tank *L* is the wash-water tank *E*, which is connected to said tank by means of the pipe *O''*, through which the wash-waters from said screen-tank can be transferred to said wash-water tank.

R is the vacuum-pump, having a connection at *p'* with the bottom of the screen-tank and discharging into the catch-all or tank *Q*, from which the wash-water can be transferred to the wash-water tank *E* by means of the service-pump *f* and pipes *f'' f'''*.

Procedure of Causticization.—The operation of the plant is as follows: Water is run into the converting-kettle *A* and brought to a boil therein, and then a given quantity of soda-ash is put into the said converting-kettle and the boiling continued till the complete dissolution of the ash, making a solution of about 12° Baumé. Then small portions of lime are added gradually through the medium of the lime-basket *j*, placed at a point in the converting-kettle so that such basket will be suspended partly in the liquid. The boiling (with open steam) is continued and lime added for the conversion of the soda-ash into caustic soda, which can be determined by testing in any suitable way. When the liquid is thoroughly causticized, as indicated by the suitable test referred to, the steam is shut off and the whole allowed to settle for a time, after which the clear liquid is then drawn off from the converting-kettle by means of the pipe and elbow *f*, either by gravity or being pumped and run to a suitable tank or other receptacle *F* for storing the liquor, from whence it is delivered direct to the soap-factory or refinery, or if not of sufficient density it is then run to the evaporator *G*, which can be of any suitable construction for concentrating it to the desired density. The eduction-pipe from this evaporator connects, preferably, with a catch-all *G'*, by means of which moisture mechanically entrained with the vapors may be arrested and conveyed back to the evaporator. There will then remain in the converting-kettle *A* mud composed of calcium carbonate entangled with caustic-soda lye, which mud is drawn from the converting-kettle through either or both of the outlets *c* or *d* in the bottom of said kettle and run into the screen-tank *L* and allowed to settle, and the liquor above the lime-mud or residue is drawn off through a suitable pipe *O''* siphonically or by means of a pump, or can be run off by gravity through an outlet or outlets *O'''* on the side of said screen-tank, and is run to the same tank *F* as the clear liquid that was previously drawn off from the converting kettle, or if of insufficient strength it may be run to the tank or receptacle *E*. Then water is run into the screen-tank and the mass brought to a boil by the injection of steam through the perforated steam-pipe or coil, after which the mass is again allowed to settle, and the resulting clear liquid that then settles above the lime-mud is decanted or drawn off in the same manner. Then more water is added to the mud in the screen-tank and the same operation repeated until the clear liquid that settles in said tank shows "0" Baumé when tested with a regular alkali hydrometer. Then the remaining liquid with which the mud is saturated is drawn therefrom by means of applying a vacuum below the screen, the liquor being drawn by said vacuum into a suitable receiver, preferably a cylindrical vessel *Q*, and the liquor thus collected in the cylindrical vessel is then run to join the other wash-waters that have been drawn from the screen-tanks by other means. Such of these wash-waters as have been run from the screen-tank *L* to a receptacle *E*, separate from the storage-tank from which the refinery, soap-house, or evaporator is supplied, are used in the converting-kettle for dissolving the next charge of soda-ash, such water being added in addition thereto as may be required, which additional water is preferably a water of condensation from the steam-coils or heating-chamber of the evaporator, which water of condensation shall have been run to the tank *I* or other suitable receptacle.

The Manufacture of Sal-soda.—Sal-soda was at one time an article manufactured to large extent by soap-manufacturers in this country when the supply of alkali was obtained entirely from English sources. The development of the alkali-industry in this country, however, effectually put a stop to its manufacture in isolated plants. In recent years its manufacture has been taken up to some extent from the soda-ash obtained directly from the alkali-manufacturer. Those engaged in the business are able to meet the demand in their respective localities and make a profit, due to difference in freight between soda-ash and sal-soda.

The manufacture is simple, requiring but little outlay for capital and labor, the chief requisite being space and suitable natural conditions of temperature. The process is carried out in four successive stages, viz.,

(1) Solution of high-test alkali in water.
(2) Purification of solution.
(3) Crystallization of the sal-soda.
(4) Separation of crystals from mother-liquor.

The solution of the soda-ash is best effected in an apparatus similar to the one shown in Fig. 28. The dissolver is also provided with a steam-jet not shown in the figure.

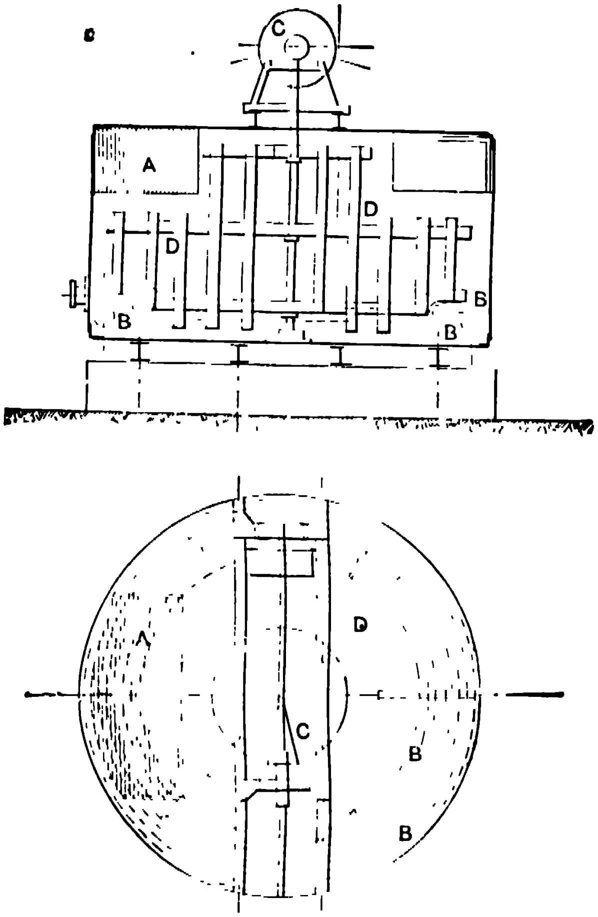

FIG. 28.—Soda-ash Dissolving-tank.

The alkali should be dissolved in the following manner: As soon as the water in the dissolver begins to boil, the ash is put on a strong wire sieve, the bottom of which is immersed just below the surface of the boiling liquid; a basket of thin iron rods placed parallel answers well for this purpose. The alkali must be gradually added, and the solution well agitated; this, as well as the employment of the sieve, is necessary on account of the caking properties of soda-ash. One part of soda-ash in two parts of water at the boiling temperature will show a density of about 34° Bé. Calcined Glauber's salt—5 to 10 per cent of weight of ash—is added at this stage.

The dissolving-tank is fitted with discharge-pipe at the bottom for drawing off the sediment and impurities, and with a second pipe, about four inches from the bottom, for running off the mother-liquor into the crystallizing-tanks by way of a 1½-inch pipe leading into the cellar, and emptying into an iron channel, which can be directed to deliver into any of the vats at will.

In the following table are shown analyses of low and high test soda-ash and of soda crystals.

TABLE XII.—ANALYSES OF SODA ASH AND SODA CRYSTALS.

	Soda Ash.		Soda Crystals.
	48%.	58%.	
Sodium carbonate, Na_2CO_3	60.64%	98.72%	34.22%
Sodium sulphate, Na_2SO_4	1.35%	20%	2.54%
Sodium hydroxide, NaOH	1.20%	.	.10%
Sodium chloride, NaCl	28.34%	54%	.27%
Calcium carbonate, $CaCO_3$	traces	13%	
Magnesium carbonate, $MgCO_3$		04%	
Iron oxide, Fe_2O_3	1.12%	04%	
Alumina, Al_2O_3		01%	
Silica, SiO_2		09%	
Moisture, H_2O	43.6%	20%	62.84%

Glauber's salt crystallizes in large, colorless monoclinic prisms and before the advent of high-test soda-ash, crystals of this character could be obtained from the soda-ash of those days, owing to the presence therein of Glauber's salt. High-test soda-ash will crystallize in the smaller crystals peculiar to sodium carbonate and tend to be discolored by the enclosure mechanically of the mother-liquor. The addition of anhydrous Glauber's salt, in quantities regulated by experience, tends to the formation of larger, and, if proper conditions are observed, more colorless crystals. In accordance with the following equation:

$$Na_2CO_3 + \underset{180}{10H_2O} = Na_2CO_3,10H_2O,$$

100 parts of sodium carbonate will yield 269.81 parts of sal-soda. In accordance with the following equation:

$$\underset{142}{Na_2SO_4} + \underset{180}{10H_2O} = Na_2SO_4,10H_2O,$$

100 parts of anhydrous sodium sulphate will yield 226.76 parts of the crystallized salt, the yield being less than with soda-ash.

With a permanent demand and a knowledge of the localizing influence of freight rates, the margin possible in a business of this character can be readily estimated.

Purification of Solution.—This may be done most economically in settling-tanks, but on a large scale the filter-press is commonly employed. The chief impurities are insoluble metallic oxides.

Crystallization.—The crystallizing-tanks are of various sizes and, to insure durability, are preferably of iron. Brunner, Mond & Co. recommend a tank 6′×10′×2′. The tank is usually provided with slats of wood or iron, immersed in the liquor to hasten crystallization. The pans are allowed to stand for a week or ten days when the mean temperature of air is about 15° C.; in warm weather longer time must be allowed.

The maximum crystallization has been obtained when the mother-liquor indicates a density of 20° to 22° Bé. The mother-liquor is drawn off and after clarification by settling or filtration is added to the dissolving tank again, sufficient water being mixed with it to dissolve a fresh charge of soda. The crystals are freed from adhering mother-liquor by the addition of a small amount of water in a hydro-extractor, or to the crystals suspended in a crib over the crystallizing tank. The wash-water is transferred either to the dissolver or to the crystallizing-tank. There need be no waste and the theoretical yield should be very closely approximated.

It will be of interest to note the following peculiarities of solutions of sodium sulphate and sodium carbonate: The crystallization of the former salt with 10 molecules of water is dependent upon the temperature of the solution being below 33° C. at the time it is deposited. If a saturated solution is cooled to a point below 33°-40° C., the salt separates with water of crystallization. When the crystallized salt is heated to 33° C. it loses part of the water of crystallization. In solutions below 33° C. the salt $Na_2SO_4,10H_2O$ is probably present. Above this temperature the compound suffers decomposition with the formation of anhydrous and less hydrated salts.

Sodium carbonate behaves similarly to sodium sulphate; its maximum solubility lies between 33° and 70° C., the solubility decreasing above the latter temperature.

Potassium Carbonate and Potassium Hydrate.—Potassium carbonate was the first alkali used in the soap industry. It was converted into the caustic form by primitive causticization with lime, whence the liquor obtained on settling the mixture was used for saponification. The potash soap thus made was grained with salt, whereby an interchange of alkali occurred with the formation of a hard mixed soda and potash-soap. With the clearing of American forest lands the chief commercial source of potash was transferred from the ashes of kelp and barilla to that of wood. Potassium carbonate is also obtained to some extent from the calcined suint of wool-grease and from the residue left on the distillation of beetroot molasses. Relative to the manufacture of potash from wood-ashes, J. N. Lloyd * states: "What is potash? Few would hesitate to answer this question, and yet the commercial substance is not as easily defined as might be imagined. Potash should be the residue that is obtained by lixiviating the ashes of wood, causticizing the liquor thus obtained with lime and evaporating the liquor to dryness. The calcined residue constitutes commercial potash and is a mixture of insoluble matter, 1.5 to 3 per cent; sulphate of potassium, 5 to 15 per cent; chlorides of sodium and potassium, 5 to 10 per cent; carbonates and hydrate of potassium, 80 to 95 per cent."

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* Before the American Pharmaceutical Association.

Although considerable potash of this character yet comes on the market, chiefly from Canadian sources, the chief commercial supply of the world is the Stassfurt deposits of northern Germany.

According to the best authorities, the Stassfurt deposits of potash had their origin in past geologic epochs by the isolation of a part of the sea, the waters of which were heavily charged with potash salts. This isolation was at first incomplete, and as the evaporation of the enclosed waters took place they were supplied by small estuaries leading to the ocean, and by a continuation of this process the percentage of saline matters in the waters rapidly increased. In those ages the climate of Europe was still tropical, and the rate of evaporation was therefore much more rapid than at the present time. The less soluble materials, such, for instance, as gypsum, naturally were the first deposited, and as common salt was the most abundant mineral ingredient, these deposits of gypsum were covered with thick layers of rock salt as the next deposit. This layer ultimately reached a thickness of 3,000 feet, and it is stated by geologists that it required at least 13,000 years to form it. The deposit of rock salt is not continuous, but is broken occasionally with lamellated deposits of sulphate of lime and, toward the top of the formation, by layers of the mineral called polyhalite, which consists of the sulphates of lime, potash and magnesia. Above these deposits are found other layers containing the mineral kieserite (sulphate of magnesia). Above the kieserite line the chief deposits of potash salts consist mainly of the mineral carnallite, composed of chloride of potash and chloride of magnesia. The carnallite deposit is from 50 to 130 feet in thickness, and yields the most important quantity of the crude potash from which the manufactured salts of commerce are made. Above the layer of carnallite is found a covering of clay which is almost impervious to water, and it is this water-tight covering which has preserved the soluble mineral deposited under it from subsequent solution in percolating rain-water. Had it not been for this protection these deposits of potash, now of such great industrial importance, would long ago have been washed away and lost.

Again, above the clay is another stratum of sulphate of lime, showing that after the deposit of the clay the original process of the deposition of mineral matter was continued, since above the sulphate of lime is found again a layer of rock salt; but this rock salt is of a purer quality than that of the first layer mentioned. The deposits are completed by another layer of sulphate of lime and of impervious clay capped by sand and limestone, which crop out at the surface of the soil.

The perpendicular distance from the surface of the lowest of the Stassfurt salt deposits is about 5,000 feet, while the horizontal extent of the bed is from the Harz Mountains to the Elbe River in one direction and from the city of Magdeburg to the town of Bernburg in the other.

The composition of the principal minerals occuring in these deposits is given in the following table:

TABLE XIII.—COMPOSITION OF MINERALS OF THE STASSFURT DEPOSITS.

Constituent.	Kainite.	Carnallite.	Polyhalite.	Krugite.	Sylvinite.	Kieserite.	Schonite.	Potassium Sulphate.		Potassium Magnesium Carbonate
								High Grade.	Low Grade.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Potassium sulphate...	21 3	..	28.90	18 60	6 97	...	50 40	97.20	90.60	
Magnesium sulphate .	14.5	12.10	19.93	14.70	4 80	21.50	34	.70	2.70	
Magnesium chloride. .	12.4	21.50	....	. . .	2 54	17 20	.	.40	1	
Potassium chloride. . .	2	15.50	..	.....	30 55	11 80	..	.30	1.60	
Sodium chloride......	34.6	22.40	.....	1.50	46 05	26.70	2 50	.20	1.20	
Calcium sulphate.....	1.7	1.90	45.18	61	1.80	.80				
Water.............	12.7	26.10	5.99	4.20	7.29	20.70	11.60	.70	2.20	25
Alumina...........	.8									
Undetermined........	.....	.50								
Insoluble....	.....	.....	.....	...	.....	1.30	.....	.20	.30	
Potassium carbonate..	....	....	.....	.....	.....	....	.....	. .	. .	35–40
Magnesium carbonate	.....	.....	.....	....	....	.....	....	...	...	33-36

The potassium chloride is prepared by leaching the carnallite, or other crude salts containing potassium chloride, either with hot water or a hot concentrated solution of magnesium chloride in such proportions as to dissolve the potassium and magnesium chlorides, but not the common salt. On cooling this solution to 70° C. and allowing it to remain for some time, the potassium chloride is deposited in a crystalline form. A second crop of crystals is also obtained by cooling the mixture to usual temperatures. On concentrating the residual mother-liquor, another crystalline deposit, consisting of mixed potassium and magnesium chlorides, is obtained, which can be added to the crude salt and re-treated as above. The crystals of potassium chloride obtained by the first two crystallizations are washed, drained, dried, and packed for shipment. By repeated evaporations, crystallizations, and resolution, about 85 per cent of the potassium chloride is finally obtained, only about 15 per cent being lost in the waste waters. Potassium carbonate is made from potassium chloride after the general manner of the Le Blanc process. Caustic potash is made from potassium carbonate by causticization with lime as is done in the manufacture of caustic soda. Caustic potash obtained from this source is of more uniform quality and of a higher degree of purity than that obtained from wood-ashes, though it is contaminated with variable proportions of the chloride, sulphates and carbonates of sodium and potassium and with caustic soda. By electrolytic process commercial caustic potash is now prepared, analyzing 85–90 per cent actual potassium hydrate. Caustic potash is to be distinguished from caustic soda which it surpasses in chemical activity by forming soft soaps with fats and oils, which for many purposes, as in the textile industry, are superior to soda soaps. Beyond the manufacture of high-grade textile soaps, of certain pharmaceutical soaps and as a partial ingredient of shaving-soap, caustic potash is of limited application in the soap-industry, being displaced by the cheaper sodium compounds. Carbonate of potash is likewise displaced by the corresponding sodium salt.

Salt.—In soap-boiling salt, sodium chloride, serves as a purifying agent which property is based upon the insolubility of soap in aqueous solutions of salt. Common salt is widely distributed in nature as the mineral Halite. The early process of manufacture consisted in its production from sea-water, either by exposure to evaporation under the sun's rays, or sometimes by boiling in pans or kettles until the dissolved salt was deposited. About the close of the eighteenth century the manufacture was begun from brine obtained from natural salt-springs, following the same crude processes used in the manufacture of salt from sea-water.

At the present time, however, comparatively little salt is made from sea-water, or brine from natural springs, and some of that so obtained is evaporated by artificial heat. Nearly all the evaporated salt is now obtained by sinking wells to the salt body, pumping fresh water into the wells, and withdrawing the brine after it has become well saturated. Practically all the product from natural salt water, by solar evaporation, is made along the shores of San Francisco Bay in California, and Great Salt Lake, Utah. With these two exceptions the evaporated product is almost entirely obtained from deep wells.

Salt manufactured by artificial heat is made in kettles, open pans, vacuum pans, and grainers, the heat being applied either directly or by steam. In blocks where kettles or open pans are used, the heat is usually applied directly and the brine is boiled. In grainers and vacuum pans steam-heat is used. The grainer process is essentially American; the brine in this process is evaporated from rectangular vats about 12 inches deep, in which are suspended coils of pipe carrying either live or exhaust steam, according to local conditions. The brine is usually kept agitated mechanically, so that the salt which is formed on top will be broken up and precipitated. In some instances the salt is removed from the bottom of the pan by mechanical scrapers; in others, hand labor is employed. The grainer process seems to be the most popular method in the United States and most of the finer grades of table and dairy salts are produced either by this or by the vacuum-pan process.

Solar salt is made in vats or ponds, covered and uncovered. At Syracuse, N. Y., the ponds are supplied with movable covers. No covers are used in Utah or California, as the operations in those states continue only during the dry season.

Rock salt is mined and prepared for use in the states of New York, Kansas, Louisiana, and California. It is now produced in greater quantities than solar salt.

The impurities, chiefly iron oxide and the chlorides and sulphates of lime and magnesia, are removed by subjecting the solutions of crude salt to processes of settling or precipitation with suitable reagents and fractional crystallization. The solution thus purified is concentrated by either solar heat, by direct fire as in kettle and pan methods, by the steam-grainer system, as in steam-pans, steam-kettles, etc., or by steam under reduced pressure, as in the vacuum system. In recent years the use of recovered salt from waste soap-lye has reduced the consumption of this article in the soap industry. The salt is commonly received in bags of 200 pounds each.

Preparation of Brine.—Salt in the dry state is used on the stock and rosin changes of settled soap. If required on the strengthening change it should invariably be in solution. The brine may be made up as required conveniently in a tank mounted on a truck. To reduce labor and to provide a steady supply one of the best methods in use consists in allowing water to percolate through a body of salt. Take a large water-tight barrel or cask (Fig. 29) and fit a false bottom or wooden grating 6 or 8 inches above the bottom—this can be made of strips of wood about an inch square, and placed not over one-half inch apart. This false bottom should

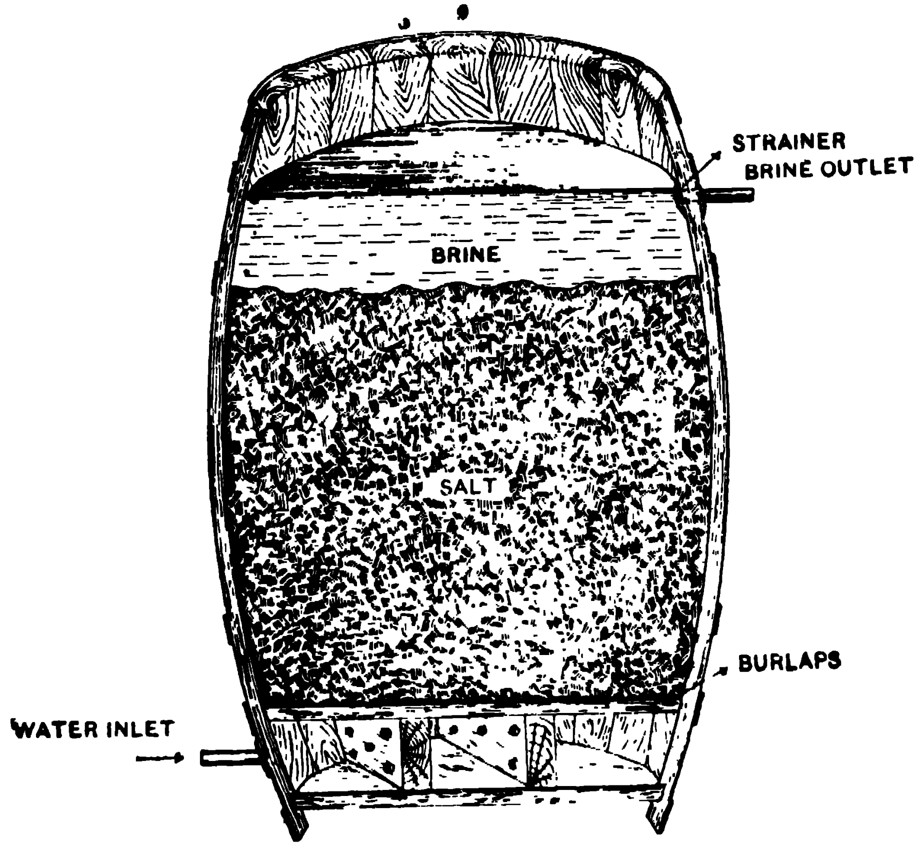

FIG. 29.—Percolating-tank for Brine.

be supported by two strips of board each 6 inches in width, placed on edge and nailed to the bottom. These boards should have several holes bored near their bottoms to permit a free passage of water. The water inlet should be below the false bottom.

A single thickness of burlap should be stretched across the top of the false bottom and tacked to the sides of the barrel. The outlet pipe for the brine should be 4 or 5 inches below the top of the barrel; the water is supplied at the bottom from a convenient hose or faucet. The supply-pipe should be of about 1¼ inch diameter, and the outlet pipe about 1½ inch inside diameter. If it is necessary to make brine faster than can be accomplished with one barrel, fit up two or more extra barrels.

TABLE XIV. FREEZING- AND BOILING-POINTS OF SODIUM-CHLORIDE SOLUTIONS. LUNGE AND NAVILLE.

Per Cent NaCl in Solution	Freezing-point °C.	Boiling-point °C	Per Cent NaCl in Solution	Freezing-point °C	Boiling-point °C.	Per Cent NaCl in Solution.	Freezing-point °C.	Boiling-point °C.
0	+0	+100	....		.....	20	−14 44	+105 46
0.1	−0 08	100 02	9	−6 72	102 11	20 5		105.63
0.2	0.15	100 04	9 5	7 08	102 24	21	15 11	105 81
0.3	0 23	100 06	10	7 44	102 38	21 5	.	105 98
0 4	0 31	100 08	10 5	7 80	102 52	22	15.78	106 16
0.5	0 38	100.10	11	8 16	102 66	22.5	. .	106 34
1	0 76	100.21	11 5	8 52	102 80	23	16 45	106 52
1 5	1 14	100 31	12	8 88	102 94	23 5		106 70
2	1 52	100 42	12 5	9 23	103.08	24	17 11	106 89
2.5	1 90	100 53	13	9 59	103 23	24 5		107 08
3	2 28	100.64	13 5	9 94	103 38	25	17 77	107 27
3.5	2.66	100 75	14	10 29	103 53	25 5		107 46
4	3.03	100.87	14 5	..	103.68	26	18 42	107.65
4.5	3 40	100.98	15	10 99	103 83	26 5	.	107 84
5	3 78	101 10	15 5	.	103.99	27		108 04
5.5	4 14	101 22	16	11 69	104 14	27 5	.. ..	108 24
6	4 52	101 34	16 5	.	104 30	28	.	108 43
6.5	4.89	101 46	17	12 39	104.46	28 5	.. .	108 43
7	5 26	101 59	17 5	.	104 63	29	. .	108 63
7.5	5 62	101 72	18	13.07	104 79	29 4	. .	108 83
8	5.99	101 85	18.5	.	104 95	30	.....	108.99
8.5	6 35	101 98	19	13 76	105 12			
.... .	.. ..	....	19.5		105 28			

To make brine—fill the barrel above the false bottom with salt, and turn on the water. The salt will dissolve rapidly, and more must be shovelled in on top. The barrel must be kept full of salt, or the brine will not be of full strength. No stirring is necessary. Keep skimming off all waste matter rising to the top. The brine outlet should be provided with a strainer of some kind to prevent chips, etc., from running out with the brine.

Filling Materials.—The incorporation of foreign material, either detersive or inert, with soap, may be said to be coexistent with the use of mechanical crutchers and originated in the effort to cheapen the cost of manufacture. The most important filling agents are soda-ash and sodium silicate. The first use of soda-ash in soap is attributed to Van Haagen, of Philadelphia, shortly after the Civil War. The first use of sodium silicate was by Christopher Thomas & Bros., of Bristol, England, in 1856. It was not, however, until 1862 that its value was officially and publicly recognized, when at the international Exposition in that year in London, a prize was awarded to Wm. Gossage & Sons for their samples.

By the use of reasonable proportions of solutions of soda-ash and sodium silicate, the quality of a laundry soap is greatly improved. The soap is made more durable from the hardness produced by the crystallization of the salts; also, the rapid drying of the soap is prevented. The detergency of these additions justifies their use, and in reasonable proportions they constitute no adulteration. Whether or not a soap may be called adulterated is determined primarily by the price at which it is sold in a competitive market. The demand of the public is mistakingly for cheap things.

Soap-manufacturers are compelled to turn out a product that looks well and feels well and costs little. The manufacturer satisfies a mistaken judgment that considers appearance and quantity, and that disregards commendable efforts to maintain a standard of quality.

Silicate of Soda.—Silicate of soda as used in soap-manufacture is a colorless, viscid aqueous solution of the product of the combination of pure sand with either soda-ash or caustic soda. The product is essentially a glass differing from the hard, vitreous form in that the soluble alkalis of sodium or potassium replace the insoluble lime, giving rise to a product soluble in water; hence the term "soluble glass." Combination of sand (silica, SiO_2) with alkali may be effected in either a reverberatory furnace, in which case soda-ash (Na_2CO_3) is used; or in a digester under pressure, in which caustic soda ($NaOH$) solution of varying density, according to the density of the product desired, is used. The proportions of silica and alkali used depend upon the character of the desired product and their variation gives rise to the various grades of the product used in the arts. Made by the dry process, in a reverberatory furnace, the fused product is discharged and cooled and may be shipped in the transparent, hard and brittle form and solution effected at its place of use. Solution of hard silicate of soda can be effected commercially only under steam pressure, for which purpose a digester is used. This is filled with the hard silicate and a proportion of water, determined by the density of the solution desired, and steam up to 80 pounds pressure admitted. At the end of the dissolving period the product at about 30° Bé. is discharged to a settling-tank, or directly to evaporators and concentrated to any desired density. Made by the wet process, in a digester, the product for many purposes may be discharged at the desired density, but for densities greater than 30°-32° Bé. subsequent concentration is necessary.

The degree of concentration of commercial water-glass varies considerably. It is generally determined by the hydrometer, but this instrument alone affords only a one sided indication of the true character of the solution, since the composition as well as the specific gravity of the silicate itself is subject to considerable variation. Of the ingredients, silicic acid and alkali, the former has a much lower specific gravity; consequently an increase in the contents of the silicic acid decreases the specific gravity of the compound, as well as of its solution of a given concentration. Therefore the hydrometer can be relied on to give exact indications only when the composition of the silicate is also known.

A density of 40° Bé. is used in soap-manufacture.

When silicate of soda solution is concentrated it loses a part of its silica after a certain stage of concentration. The further concentration is carried the more silica it loses; hence, dense solutions are rich in alkalis and weak solutions richer in silica. This, however, only with glass having a base of soda, which can be decomposed much more easily than potash glass.

The solubility of silicate of soda is a resultant of the proportions in which the acid and basic ingredients are combined; the solubility increases as the proportion of alkali increases, and diminishes as the proportion of silica increases; thus the alkali silicate is more soluble than the neutral silicate. Certain grades of silicate will absorb strength up to one-third of its weight of 35° Bé. lye.

Silica is of weak acid properties and is displaced from its soda compound by carbonic acid of the atmosphere. Jellying of silicate of soda solutions, resulting from exposure to the atmosphere, may be overcome by mixing sufficient caustic lye to combine with the silica.

The consistency of silicate of soda of the density commonly used, viz., 40 Bé., lends itself well to incorporation with fluid soaps. Its chief effect on soap is to harden it on ageing, to reduce the tendency of alkaline salts to effloresce, and to a certain degree to overcome the sticky feel common to rosined soap. While mixing well with soap itself it aids in the incorporation of other material. Its detergent effect in soap is slight if any when the latter is fresh; but on ageing its effect is to reduce the detergency of soap. It tends to increase the smoothness and gloss of settled soap and to destroy the natural texture. In the use of sodium silicate as a filling agent the opinion of C. F. Cross is of interest in this connection: "The solution of sodium silicate is inferior in lathering properties, its detergent action is not aided, as in the case of soap, by the softening action of the acid constituent upon the surface to be cleansed; and lastly it cannot be brought into a form so universally convenient as that of soap, existing only as a glassy solid, or a viscous liquid. Then it can only be used in a state of solution, or when incorporated with a sufficient proportion of soap to allow the physical properties of the latter to predominate. These considerations will enable us to appreciate the right side of the 'silicated soap' question. The wrong side consists not in the production by means of the silicate of a detergent of lower quality, but in not selling it at a proportionally low price. A silicated soap is an advantage to commerce, for there is much work which it can perform as well as, if not better than, soap of higher quality. But let there be a fair division of advantage between manufacturer and consumer." *

With settled rosined soap its use ranges from 25 to 50 pounds per frame and with cold process soap up to 100 per cent of the weight

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* Lecture on Soap. International Health Exposition. London, 1884.

of glyceride stock. When used to a large extent in cold-process soap, additional lye should be used above that required for the stock to neutralize the silicate. The lye and silicate may be added separately. Common precedure is to add lye and silicate successively to the stock.

Borax.—The natural source of borax is the mineral borocalsite which consists chiefly of borate of lime. The process of treatment depends upon the reaction of borate of calcium and sodium carbonate, which, when added, form borax and carbonate of lime.

The crude borate of lime is first passed through rock breakers and is then ground to the fineness of flour by means of rolls and burr stones. It is then, with a small proportion of carbonate of soda, thrown into a digester, where, under heat, pressure, and agitation, the existing affinities are completely divorced. The carbonic acid unites with the lime, which yields boracic acid, the latter with a small portion of soda, and the result is borax in solution. The liquor is then drawn off into tanks, where the borax in crystalizing attaches itself to small steel rods and hooks. The sediment contained in the mixing tanks is composed largely of sand and dirt with considerable borax mixed. The deposit is passed through a filter press, which presses the dirt and allows the borax liquor to pass away to be utilized again. Repeated over and over again, the last remnant of borax is finally secured by this process.

The most productive borax mines are in California, although the mineral is widely distributed. Borax, contrary to popular repute, is of limited use in the soap industry. It is mildly alkaline and possesses valuable detergent and preservative properties when used alone. There is no use in the soap industry for which it can not be replaced by the more strongly alkaline and detergent carbonate of soda.

Talc.—Talc is a hydrated silicate of magnesia and is recognized by its extreme softness and unctuous feel. It is a common mineral and in small quantities is widely distributed.

Talc is employed in the arts in two distinct forms—as powdered or "flour talc," and as sawed pieces of various sizes and shapes, for which latter form the soapstone or steatite variety is used. Commercially the name talc is usually applied to the fibrous and foliated varieties, which are the purer forms, and the name soapstone confined to the massive varieties. The former is the more valuable, and the greater part produced is used in the manufacture of paper.

While talc is used to some extent as a cheapening agent in laundry-soap of inferior grades and in soap-powder, it finds more general use as a filler in milled toilet-soap. Its presence is a positive adulteration of which the character of the material admits to a high degree. It is sifted upon the soap during the process of milling. It not only increases the yield in virtue of its own weight, but also by its marked hygroscopicity whereby a larger amount of water than would otherwise be practicable can be retained. It is the least dangerous filling, being entirely inert as a detersive agent. It reduces the lathering power of the soap, and when present in large amount, tends to make the soap cracky as it comes from the plodder. Its presence in the soap admits of a nice smooth product in pressing and retains the form and fresh appearance of the bar on aging.

Its presence in any soap tends to impart a cloudy, turbid appearance, especially in laundry-soap; in milled soap, however, this character is least objectionable.

Mineral Soap-stock.—Mineral soap-stock is a trade-name applied to a mixture of hydrocarbons obtained as a by-product in the manufacture of paraffin, both of which are among the highest-boiling portions of crude petroleum. It is usually of a consistency intermediate to that of heavy cylinder-oil and vaseline. It is a cheapener, pure and simple, with no justification for its use in any property that it possesses. It may be added to cold-process soap to which it is claimed to impart the feel and appearance of boiled soap. It may be added in almost unlimited quantity to semi-boiled and grained soap. The sole advantage that it possesses is to "join" in the crutcher soap that tends to "open." The substance is inert and performs no function in the cleansing process.

Starch.—In the consideration of such filling material as starch, whiting or the recovered lime-waste of the causticizing kettle, talc, marble-dust, etc., we approach within the limits of direct sophistication.

CHAPTER IV.

BLEACHING AND PURIFICATION OF SOAP-STOCK.

General Considerations. Rancidity and Acidity of Stock. Relation of Quality of the Stock to Its Glycerin-content. Purification by Treatment with Sulphuric Acid. The Sulphuric Acid-Bichromate Oxidation Process. Procedure. Nature and Valuation of Fullers' Earth. The Filter-press. Bleaching with Fullers' Earth. Press-cake. Bleaching of Palm-oil by Oxygen of the Air.

General Considerations.—As a result of the development of edible uses of fats and oils and of the effort to utilize every grade of material that will promise a profit, the raw material that is available to soap and candle manufacturers is of generally inferior quality to that which was once supplied. Consequently the manufacturer's attention is directed to whatever improvement in the quality of the stock the selling price of his manufactured product permits him to buy. It may be stated that in whatever process of purification that may be applied to the raw material, there is involved no increase in the quantity of the finished product that can be made from it, but only an improvement in the appearance, chiefly the color. The nature of the process of purification used is determined by the quality and cost of the material worked upon. With tallow, e.g., at 5 cents per pound and 74° caustic at $1.85 per hundredweight for 60° grade, the cost of the process employed must bear a very small relation to the cost per pound of tallow. It requires but a consideration of this fact to demonstrate the impracticability, however efficient they may be, of the majority of bleaching processes that are so frequently offered.

Rancidity and Acidity of Stock.—The material to be removed from soap-stock may be divided into two general classes, viz., substances producing color and substances producing odor. Frequently the color and odor may arise from the same substance as the fermenting albuminous matter of inferior tallow. The color arises from the presence of non-glyceride matter, while the odor may arise from the decomposition of either or both the glyceride and the non-glyceride matter.

When fats and oils possess an unnatural odor they are termed rancid. Rancid stock is characterized by a high percentage of free fatty acids. We must distinguish between rancidity and free acidity. Acidity of fats and oils results from the decomposition of the glyceride and need involve no other deterioration than loss of glycerin. Rancidity results from the oxidation of the liberated fatty acids and of the non-oleaginous foreign matter. Conditions favoring rancidity induce acidity and the two states may develop side by side, but the former condition with animal stock becomes pronounced only with the presence of a large proportion of albuminous matter and moisture. With vegetable stock as cocoanut-oil rancidity ensues chiefly from the oxidization of the easily decomposable fatty acids of low molecular weight; with palm-oil, however, which is similar in its composition to tallow, rancidity with the accompanying high free acidity results from the primary fermentation of non-oleaginous matter in the presence of moisture.

All animal and vegetable stock available for soap-manufacture contains a greater or less percentage of free fatty acids, but is not necessarily rancid. Rancidity results from prolonged exposure of the stock in contact with albuminous matter, to moisture, air, and sunlight, with the presence of hydrolyzing ferments which with moisture are the initial inducing causes of decomposition.

Since glycerides in their natural state are colorless, the action of light alone is confined to the associated non-glyceride matter; moreover, it has been demonstrated by experiment that light to the exclusion of air and moisture is without effect upon commercial glycerides. Air is of influence only as it includes the action of oxygen and varies in its effect according to the composition of the glyceride; its effect being least with glycerides of saturated fatty acids and greatest with glycerides of unsaturated fatty acids; these two classes being represented respectively by tallow and linseed-oil, cottonseed-oil occupying an intermediate position. It is to be observed that perfectly dry air alone, as is true of light alone, is entirely without influence on oils and fats. By a process of elimination we arrive at the conclusion that the prime agency of decomposition is moisture. Investigation of this important subject has demonstrated the existence in commercial oils and fats of unorganized soluble ferments, or enzymes, which have the property of decomposing the neutral glyceride into free fatty acid and glycerin. These enzymes appear first in the growing seed and play an important party in the vital economy of the plant. They likewise influence the metabolism of animal cells and in both kingdoms of nature serve to explain the existence of the slight free acidity always found in even the freshest animal and vegetable fats. In the splitting up of the neutral glyceride the elements of water are absorbed and glycerin and fatty acids are set free. This constitutes the first step in the development of the state termed "rancid."

Further progress of rancidity is determined by the conditions surrounding the production of the particular oil or fat. With olein (cottonseed-oil) kept long in contact with moisture and the unstable albuminous matter of the cottonseed, the splitting up of the glyceride continues at a rapid rate with consequent deterioration of the oil; the same applies to stearin (tallow) exposed to moisture and unstable animal tissue; to butyrin, caproin, etc. (butter) exposed to moisture and equally unstable casein and likewise to all glycerides as they occur in their commercial forms.

Rancidity may then be attributed to the action of moisture in the presence of enzymes. The first stage in the development of rancidity consists in the liberation of free fatty acids, but at this stage we cannot term the oil or fat rancid, for free acidity can exist without rancidity. Rancidity, however, follows rapidly, for the influences of light and air (oxygen) favoring and intensifying the decomposition of free fatty acids, soon become operative, but which alone, as stated, would be without effect. The initial hydrolysis of the glycerides has been likened to the slow decomposition of granite in the course of centuries. The free fatty acids thus formed are then degraded in a secondary reaction into volatile acids of lower molecular weight which, on being oxidized by the oxygen of the air, display the disagreeable taste and smell which are comprised under the term "rancidity."

Liquid fats (oils) become rancid more readily than solid fats and the general belief, not conclusively demonstrated, that oleic acid is the principal acid set free forms the basis of the arithmetical calculation of the free acidity test, but in a series of experiments made by A. Thum* to ascertain the order, if any, in which oleic, palmitic and stearic acids are liberated from their corresponding glycerides in the production of acidity and rancidity, he found that the ratio between oleic acid and the solid fatty acid in the free fatty acids of palm- and olive-oils, is the same as that in which they exist in the neutral fat. The following causes conducive to a high percentage of free fatty acids in the various grades of tallow may be enumerated:

(1) Allowing fat to stand before rendering, especially in warm weather. After death of the animal, decomposition immediately sets in and the first step in the decomposition of tallow is the separation of the glycerides into fatty acids and glycerin.

(2) An excessively high temperature in rendering increases the amount of free fatty acids. The presence of water in the tissues and the influence of a high temperature in the rendering-tank tend to the hydrolysis of the glyceride.

(3) Rendering in closed vessels increases the percentage of free fatty acids. It is clear that there prevail here conditions that are purposely introduced and maintained in the saponification process for the manufacture of fatty acids and glycerin.

It is therefore evident that the percentage of free fatty acids is a reliable index not only of the quality of the stock used by the renderer, but a telltale upon the care which he has employed.

Relation of Quality of the Stock to its Glycerin-content.—It is important that we should clearly understand the significance of the free fatty acid percentage as an index of the amount of glycerin lost in the manufacture of tallow. Since the free fatty acid is assumed to be oleic acid, a molecule of the glyceride olein will serve the purpose of explanation. The molecular weight of olein is 884;

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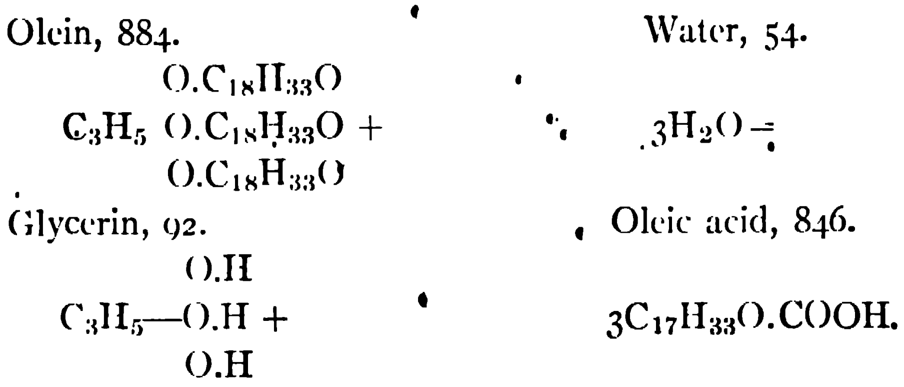
* Zeit. fur Ange. Chem., 1890, 482, 483.

the molecular weight of glycerin is 92; the molecular weight of oleic acid is 282. These numerical relations become clear on a study of the graphical representation of the olein molecule.



Olein, 884. Water, 54.

$$C_3H_5\begin{array}{l}O.C_{18}H_{33}O\\O.C_{18}H_{33}O\\O.C_{18}H_{33}O\end{array} + \quad 3H_2O =$$

Glycerin, 92. Oleic acid, 846.

$$C_3H_5\!-\!\begin{array}{l}O.H\\O.H\\O.H\end{array} + \quad 3C_{17}H_{33}O.COOH.$$

The separation of a glyceride into free fatty acid and glycerin is always accompanied by an obsorption of water as is indicated in the preceding equation; 54 parts of water combining with 884 parts of olein to form 92 parts of glycerin and 846 parts of oleic acid. Thus 100 parts of olein in saponification yield 95.7 parts of oleic acid and 10.4 parts of glycerin.

The amounts of glycerin and fatty acids theoretically obtainable from 100 parts of the tri-glycerides named are here shown:

Tri-glycerides	Chief Sources.	Yield on Saponification of 100 Parts	
		Fatty Acids Parts.	Glycerin Parts.
Stearin	Tallow and lard	95.73	10.34
Olein	Tallow and lard	95.70	10.40
Palmitin	Tallow and palm-oil	95.28	11.41
Laurin	Cocoanut- and palm-kernel oils	94.04	14.42
Myristin	Cocoanut- and palm-kernel oils	94.47	12.70
Butyrin	Butter-fat	87.44	30.46
Linolein	Linseed- and drying oils	95.21	11.58
Recinolein	Castor-oil	95.92	9.88

If a sample of tallow indicates on analysis 10 per cent of free fatty acid, there is a remainder of 90 per cent of neutral fat, indicating approximately a loss of 1 per cent glycerin on a basis of the fat, or 10 per cent calculated on the total amount of glycerin theoretically available. The extremely variable percentage of free fatty acid in tallow is patent to every soap-chemist. The statements made with reference to tallow are also applicable to bone-stock and grease, and here the loss in glycerin before the fat becomes the raw material of the soap-maker is much greater. The following table is compiled from the results of free-acidity determinations in the soap-stock mentioned. Each per cent is an average of a large number selected at random and covering the season indicated.

TABLE XV.—FREE ACIDITY OF ANIMAL SOAP-STOCK.

Soap-stock.	Per Cent Free Fatty Acids.	
	Summer.	Winter.
Tallow.	7.85	5.85
Bone-stock.	10.68	6.64
Grease.	18.03	7.49
Average.	12.18	6.66

The chief value of this generalization lies in showing the relative influence of two seasons, with the conditions peculiar to each, upon the quality of these grades of stock. It also indicates that about 9 per cent is the maximum theoretical yield of glycerin that can be accorded animal soap-stock of fair average quality used throughout the year. With animal soap-stock of fair average quality the chief commercial significance of the presence of acidity and rancidity is the loss of glycerin resulting thereby.

Tallow is composed of about 61 per cent stearin, 33 per cent olein, and 6 per cent palmitin. The varying complexity of fats and oils in their glyceride constituents, aside from their deterioration as glycerin-producing bodies in the processes of collection, rendering, refining, etc., forbids any exact statement of their theoretical percentages of glycerin. The theoretical yield of glycerin from stearin, olein, and palmitin is respectively 10.34 per cent, 10.41 per cent, and 11.42 per cent. In practice, however, there is considerable loss, as has been shown, and the figures stated are never reached. The free fatty acid in tallow, and in fact in all soap-stock, is assumed to be oleic acid and is calculated as such in the chemical method for its determination.

Purification by Treatment with Sulphuric Acid.—The procedure of soap-boiling is in itself a purifying process, the unsaponifiable matter to a varying but large degree being discharged into the waste lye. The free acidity of the stock is corrected by combination with alkali. Nevertheless sufficient coloring-matter remains to affect the color of the finished product.

With vegetable stock prone to rancidity as cocoanut and palm-kernel oils, rancidity may be entirely corrected with boiled soap by insuring complete saturation of the stock with alkali. With cold-process soaps, owing to the incomplete reaction, only the best grades of both animal and vegetable stock should be used.

Stock requiring treatment for color by the soap-maker comprises the various grades of tallow and palm-oil. Bleaching processes alone may be applied to any oil, but as a rule they are used without treatment of any kind. The installation of a bleaching-plant is determined by the quality of stock, the amount used, and the quality of soap made. Simplest treatment with sulphuric acid comprises the following procedure: A lead-lined tank provided with an open steam-coil is used. In boiling up with live steam and dilute sulphuric acid, the latter agent facilitates separation of mucilaginous matter by dehydrating it. One-fourth to 1½ per cent of 60° Bé. sulphuric acid, according to the amount of impurities present, will suffice to precipitate the mucilaginous matter. The stock may either be heated with closed steam-coils and agitated with an air-blast or heated and agitated with live steam. The acid diluted with an equal volume of water is slowly added to the stock in agitation. Cooking is continued for an hour or longer, when the contents are allowed to subside and the acid liquor is drawn off. The acid liquor, if not too foul, may be used to neutralize the alkalinity of waste soap-lye. The stock may be afterwards washed with water to remove traces of remaining acid.

The Sulphuric Acid-Bichromate Oxidation Process.—A further step beyond the dehydration and precipitation of the impurities is their direct oxidation, which more completely facilitates their removal. This process is applicable to the darker animal fats, and to palm-oil, which yields on suitable treatment a light-colored oil which forms a satisfactory partial substitute for tallow in heavily rosined soaps. The sulphuric acid-bichromate oxidation process is based upon the liberation of oxygen from potassium or sodium bichromate by means of sulphuric acid in accordance with the following equation:

$$\underset{\text{Potassium bichromate.}}{K_2Cr_2O_7} + \underset{\text{Sulphuric acid.}}{4H_2SO_4} = \underset{\text{Potass. chrom. sulphate.}}{2KCr(SO_4)_2} + \underset{\text{Water.}}{4H_2O} + \underset{\text{Oxygen.}}{O}.$$

If hydrochloric acid instead of sulphuric acid be used, chlorine is evolved from the oxidation of the hydrochloric acid in accordance with the following equation:

$$K_2Cr_2O_7 + 14HCl = 2KCl + 2CrCl_2 + 7H_2O + 6Cl.$$

If no other reaction of the nascent chlorine (Cl) ensue, it decomposes water with the liberation of oxygen, thus:

$$6Cl + 3H_2O = 6HCl + 3O.$$

Sulphuric acid preferably is used. Acid of 60° Bé. and a 25 per cent bichromate solution are the customary densities of the reagents. In the practical application of this process it is combined, as a rule, with subsequent treatment with fullers' earth and filtration. In Fig. 30 is shown in conventional form a bleaching-plant for tallow, grease, and oil.

Procedure.—The melted stock, at a temperature not exceeding 100° Fahr., is pumped to the lead-lined chemical-treatment tank. While the stock is in agitation, preferably by means of compressed air, the mixture of sodium-bichromate solution, sulphuric acid, and water is added. The proportions of the bichromate solution are 25 pounds of sodium bichromate to 100 pounds of water; the sulphuric acid is of a density of 60° Bé. The proportion of reagents used is determined by the quality of the stock and varies from ¼ to ¾ per cent of 25 per cent bichromate solution and from ½ to 1½ per cent of sulphuric acid. The mixture is diluted with not less than an equal weight of water. The oxidizing mixture is mixed intimately with the stock for a period of not less than one hour, heating being effected as desired by means of lead closed-steam coils. At the end of the acid-bichromate treatment the contents of the tank are allowed to stand to settle out the chemicals, whence they are run off either directly to the sewer or to be used to neutralize the alkalinity of waste soap-lye. If the settling period has been brief, a subsequent wash with water to remove any retained acid may be made. The stock is now transferred either by gravity, pump, or steam-siphon to the bleaching-tank, where it undergoes treatment with fullers' earth followed by filtration.

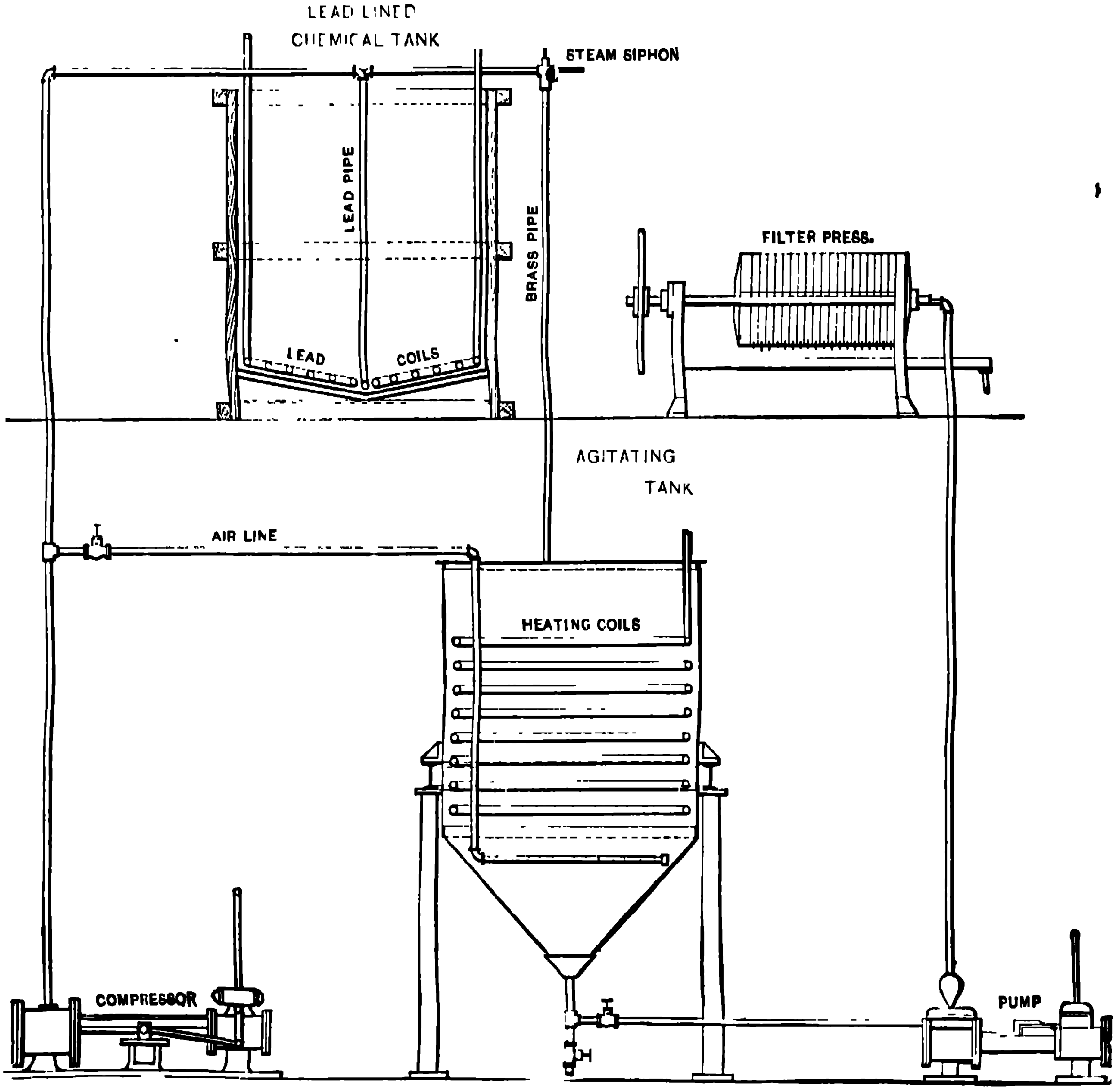


Fig. 30.—Bleaching-plant.

Nature and Valuation of Fullers' Earth.—Agitation with fullers' earth and filtration is the almost universal method for the clarification of oils. Originally, all the fullers' earth used in the United States was imported chiefly from England, but in recent years deposits of very good quality of earth have been discovered and are being worked in this country. The color, taste, physical condition, or chemical analysis does not determine the value of this body for bleaching purposes. This can be determined with certainty only by practical experiment with the oil to be bleached. When dry it adheres strongly to the tongue, but many clays, with which it may be regarded as of similar character, do the same. Unlike clay, it lacks plasticity; it has a high percentage of combined water, and a content of alumina rarely exceeding 15 per cent. Its natural color varies from brown, gray, to dark blue, due to the presence of organic matter. Fullers' earth was originally used for cleansing cloth of grease and also by furriers for cleaning furs. Its chief physical characteristics are its fine grain, non-plasticity, and that when thrown into water and broken up, it forms a somewhat flocculent mass. Its commercial value resides in its high absorbent and decolorizing power. In its preparation for market, the usual method, after mining, is to spread the material in a thin layer over a drying floor, although a more modern method is to heat it by fire in cylinder dryers. Before putting it through the dryers, it is first pulverized. In drying it assumes a white color and parts with upwards of 60 per cent of moisture. It is then sieved into various degrees of fineness, usually 100 or 120 mesh, and put in bags for shipment.

Those familiar with the use of fullers' earth know that there are great differences in the product in bleaching capacity, as well as in physical appearance. Its physical appearance is often made a gauge of its value to do work, and a sample is often accepted or rejected, merely on its color, taste or general appearance. The true measure of value of fullers' earth, however, is its capacity to do the work for which it is purchased; and of several samples of equal price, it follows that the one that bleaches the stock in the best manner, yielding the best-colored or best-flavored stock with equal amounts of earth, is the best product.

Of two samples of different price, the higher-priced product may be the cheaper to the consumer, for it may have a greater bleaching capacity than the cheaper, and leave the stock in better condition. In other words, the cost of the clay used to produce the required result per 100 pounds of stock may be actually less with the higher priced earth than with the cheaper, owing to the better quality of the former, and the smaller amount that has to be used.

This, then, is the true basis of the value of fullers' earth, viz., its actual cost to produce the best results per 100 pounds of stock.

The only classification of clays which suggests itself by which definite place can be given to the variety called fullers' earth would be based upon the consideration of the relative proportion of total bases, such as alumina, iron, calcium, magnesia, alkalis and silica, together with the proportion of water in the sun-dried clay. Very frequently the clays which have a large proportion of total bases and silica usually contain more than the average amount of water. For example, kaolin, containing from 40 to 50 per cent of silica, and from 30 to 40 per cent of total bases, will frequently contain from 10 to 15 per cent of water in the sun dried material. Where, however, the percentage of silica is very high, say 70 per cent, and the total percentage of bases less than 30, the percentage of water is usually quite low. Fullers' earth appears to be an interesting exception to this rule, as its chief chemical characteristic seems to be a high percentage of silica, which, though varying through a considerable range, frequently reaches 65 to 70 per cent, but with a total proportion of bases even as low as 20 per cent, and seldom higher than 28 per cent; while the percentage of water in the sun-dried material will range easily from 15 to 25 per cent. Approximately half of this water can be driven off by prolonged heating in a water-bath; the remainder is only to be driven off at a much higher temperature. These clays usually stick to the tongue more than other clays. When placed in water they show no plasticity, but fall apart in gelatinous flakes.

Such clays have been used for very many years, even in this country, for extracting from newly made cloth the oil used to render the wool pliable in weaving. From this fulling process the clay has taken its name.

Silliman refers to a deposit near the town of Kent, on the Housatonic River, in Connecticut. He states that: "A fullers' earth is a clay usually soapy in its feel, very absorbant of grease and oily matters; fine in its texture, so as to present no parts that shall be large and harsh enough to injure cloth or wool, mechanically, by rubbing; it should fall to powder easily in water, so as to diffuse itself through that fluid and easily mix with it and with the stuffs to which it is applied. The fullers' earth of Hampshire, England, so much celebrated, is of a greenish yellow, tolerably firm, crumbles easily in water, receives a polish from the finger nail, and is very powerfully detergent. This is, after all, the important criterion by which to distinguish fullers' earth; if it removes grease with avidity, crumbles easily in water so as to diffuse itself readily, and yet is not so coarse as to wear the fibre, it is fullers' earth. The subject is of some practical importance to this country on account of its woolen manufactures, which, although checked for the present, must eventually rise and prevail. While they are of small extent it may be better to use soap, but in very large establishments fullers' earth from its cheapness (provided it can be abundantly obtained) is very desirable.

"With respect to the existence of fullers' earth in the clay of the Kent iron bed, it appears very probable, and some of the specimens appear very like the Hampshire earth, but experiments alone can decide."

This visit of Silliman was in 1820. William Thompson found clay in a fullonica excavated at Pompeii, which was pointed out to him as the soap which the ancient inhabitants used. This was used not only by the washers and dyers, but frequently in the ordinary houses. In composition it has the general characteristics of fullers' earth.

One of the first uses for the white kaolin at Woodbridge, N. J., was for this same purpose of fulling cloth in the absence of real fullers' earth.

But the name fullers' earth has usually designated the material obtained in Surrey, England. This has been the chief source of supply for English cloth manufacturers and for export. Various attempts have been made by mineralogists to find in fullers' earth some definite mineral substance which could be referred to as distinctive and which could be found in all varieties. Thus, Dana speaks of it as being in part kaolin and partly the hydrous silicate smectite, but this has been of little value in the identification of other specimens. Even to-day the name fullers' earth is applied to any form of clay which has the characteristics mentioned above and is capable of absorbing liquids in considerable quantity, and particularly when it will act like bone-black in removing the coloring-matter from oils.

The Filter-press.—The filter-press is a machine (Figs. 31 and 36) for the separation of liquids from solid matters suspended in them. This is one of the most frequent and important operations in all the chemical industries, whether its object be that of recovering the pure liquid, or the solid matter, or both; and as the filter-press is by far the most convenient apparatus for carrying out this operation, it has found, and continues to find, very general use.

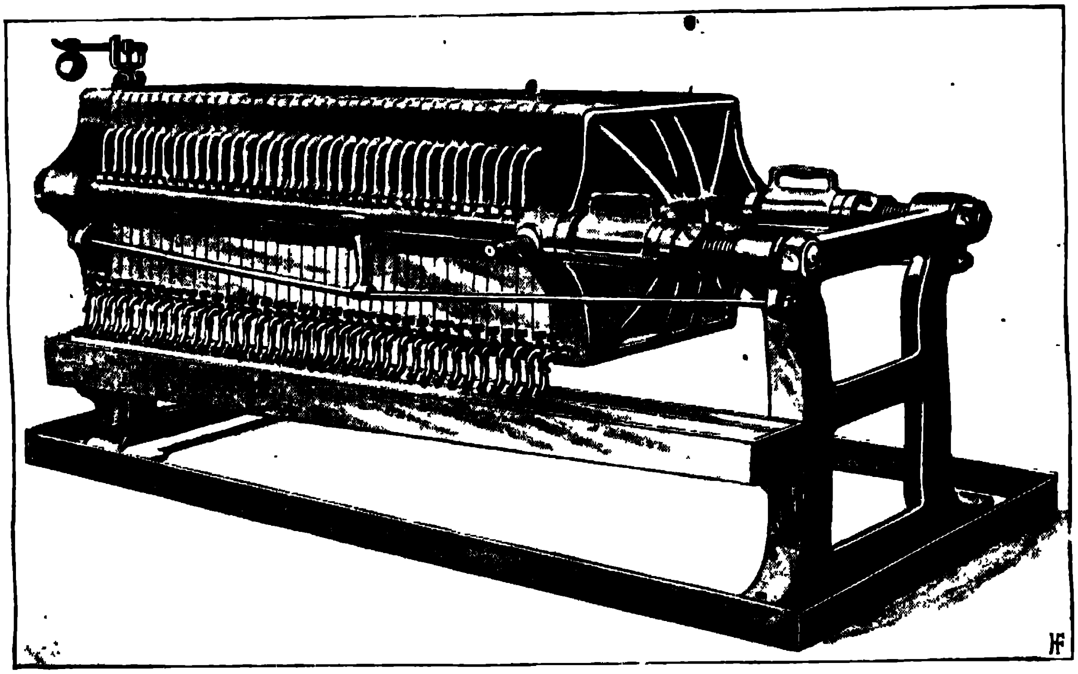


FIG. 31.—Thirty-inch Filter-press with Thirty-six Plates.

Its advantages are easily explained by its construction. A filter-press consists in principle of a number of chambers lined with filtering material, most often cotton cloth. These chambers may be formed in two ways: plates having recessed faces, the recesses in adjoining plates together forming the chamber, or flat plates alternating with frames of the desired depth. In either case the plates are provided with either vertical, horizontal, or diagonal grooves, both sides being covered with filter-cloths. The plates, or plates and frames, are clamped together between two "heads" by a strong screw lying in the axis of the press or a pair of screws working on the bars supporting the filter-plates.

All the plates and one head are provided with a hole either in the centre, side, or corner, according to the construction, and similar

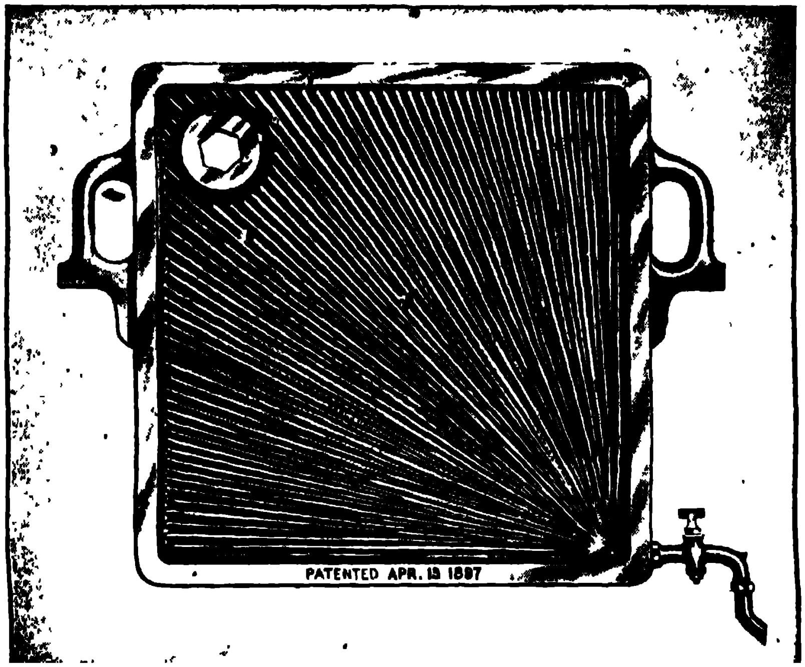


FIG. 32.—Filter-press Plate.

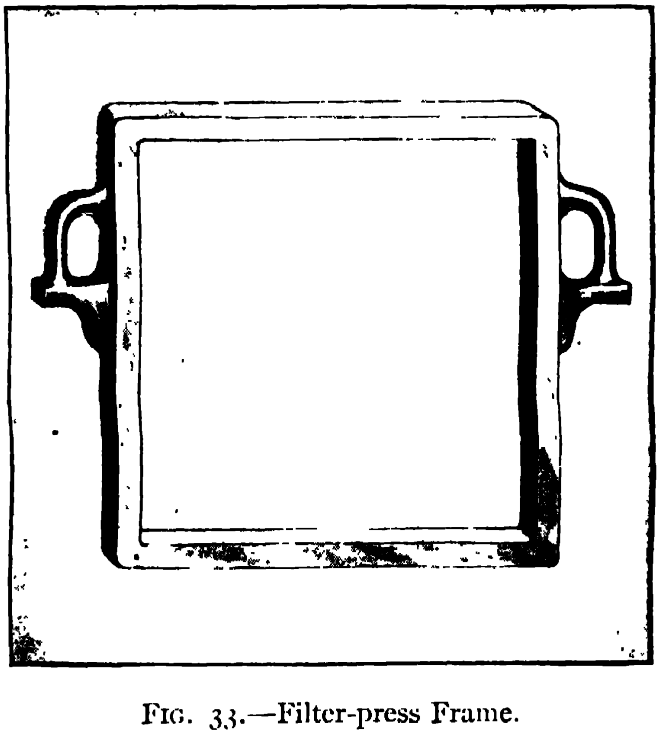


FIG. 33.—Filter-press Frame.

holes are made in the filter-cloths, a joint being made between the cloths and the plate by suitable unions. A continuous feed-channel through the whole press is thus formed.

The liquor to be filtered enters through this feed-channel under pressure, fills all the chambers, and is then forced to pass through the filter-cloths, leaving the suspended solid matter behind, which gradually fills the chamber. The clear liquor passes down the corrugations on the faces of the plates and finds its way out through a channel which connects the vertical grooves and leads into an outlet port.

Filtration continues with gradually rising pressure until the whole chamber is filled with a solid cake. As soon as this is the case, the feed is cut off, the press unscrewed, and the plates separated. The cakes of solid matter drop out, the press is screwed up and is then ready for another operation.

The efficiency of a filter-press depends upon the construction of the plate (Figs. 32 and 37); its capacity depends upon the dimensions of the plate, which may vary from 18 to 36 inches square with square plates and from 18 to 36 inches diameter with round plates, and upon the number of plates which is determined by the amount and character of the material to be filtered. The thickness of the cake may be increased within practicable limits by the insertion of frames (Fig. 33). The switch-cock and double gutter shown in Fig. 34 are very useful arrangements for separating press washings from the clear filtrate. In case of an accident to the cloth while the press is in operation, the turbid filtrate from any chamber can be returned to the tank containing the unfiltered material by directing the flow from that chamber by means of this device into the gutter emptying into that tank.

The filter-press contains the largest filtering area in the smallest space; it permits the use of high pressures and consequently rapid filtration and the formation of a hard cake, while both the liquid and the solid are obtained in the most convenient and easily handled form.

Two principal types of presses have been mentioned: one in which the plates have recessed faces, commonly known as the "recess-plate" or "chamber" press; the other, in which flat plates and frames are employed, is known as the "frame" press.

Various factors determine the choice between the two types. The chamber press is obviously cheaper, as it has only half the number of joint faces. It has the further advantage, that as soon as the press is opened and the plates separated the cake drops out. The feed-channel being large and central, there is very little risk of its becoming clogged even with heavy materials.

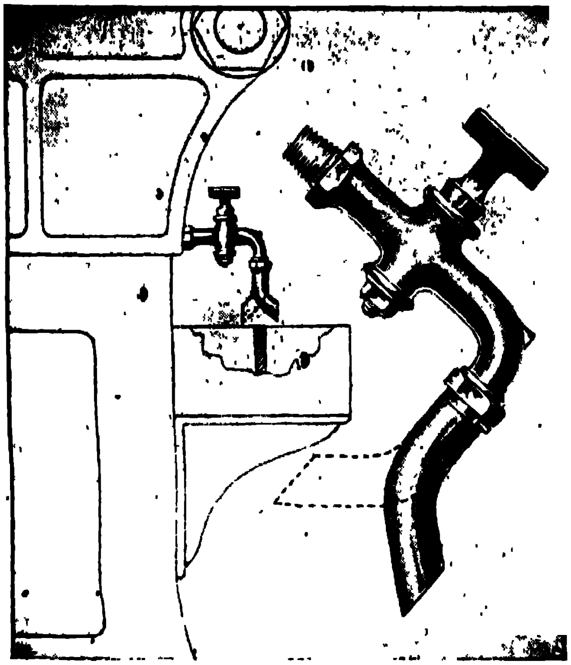


FIG. 34.—Switch-cock and Double Gutter.

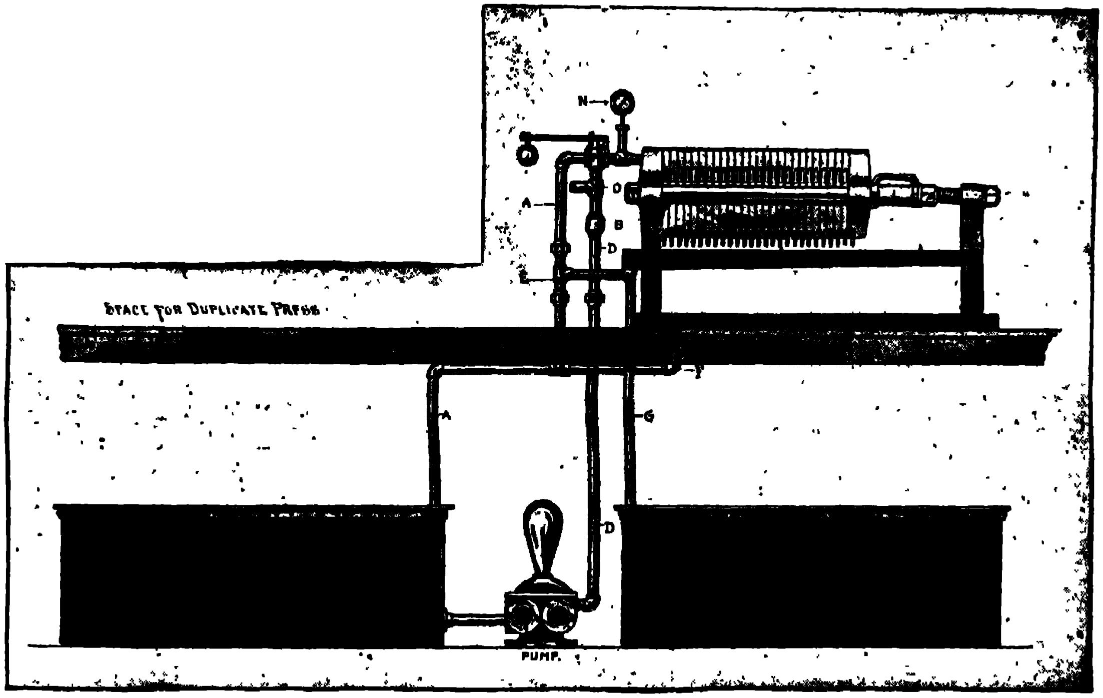


FIG. 35.—Filter-press Plant.

In the frame press the feed-channel is generally placed in flaps outside the joint of the plates and frames, so that filtering surface is gained and the cloth unions are avoided. On the other hand, the ports leading from the passage into the chamber are more easily clogged by heavy materials than the centre channel of the chamber press. When the frame press is opened, the cake remains in the frame, and this type is therefore obviously unsuited when the material does not form a solid cake.

The type of press being chosen, an important characteristic of the press still remains to be fixed, viz., the thickness of cake, or depth of chamber. This depends entirely on the material, and is a matter for experiment. As long as a cake still allows liquid to pass through it without excessive pressure, so long filtration will continue. Some materials will form cakes up to 3 and 3½ inches thick, but a more general figure is 1½ inches, which is the usual depth of the chambers of the chamber press.

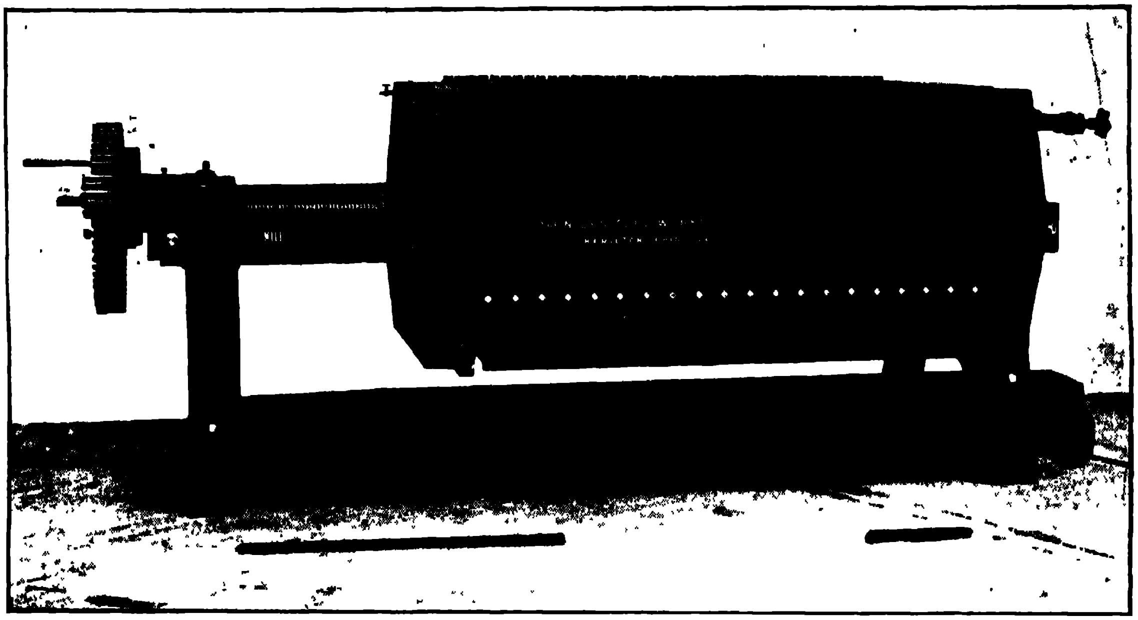


FIG. 36.—Filter-press with Steel Panel Plates.

The feeding of filter-presses is also a matter deserving close attention. As soon as the turbid liquid is admitted, a film of solid matter is deposited on the surface of the cloth, which really acts as the filtering material, the cloth being a support for it. It is essential that this solid matter should not be driven into the pores of the filter-cloth, and the pressure must therefore be put on, increased gradually, and violent fluctuations avoided. Presses may be fed by suitable pumps or by means of compressed air.

With a given type of press, a point of the greatest importance is the selection of the most suitable filter-cloth. The cloth must be of sufficiently close texture to retain all the solid particles, yet not closer than necessary, so as not to obstruct the flow of the clear liquid and retard filtration.

When the chambers are filled with solid matter, filtration ceases, the relief-valve opens and returns the unfiltered material to the tank. This stage is indicated in advance by the reduced filtrate and increased pressure on the gauge. The press is now steamed or blown out, the washings being returned to the crude tank. The nuts on the side-screws are then loosened and the blocks taken off. A man on each side of the press shoves back and loosens each cake, which drops in the pan beneath the press. After removing the cake the press is tightened up and is again ready for use.

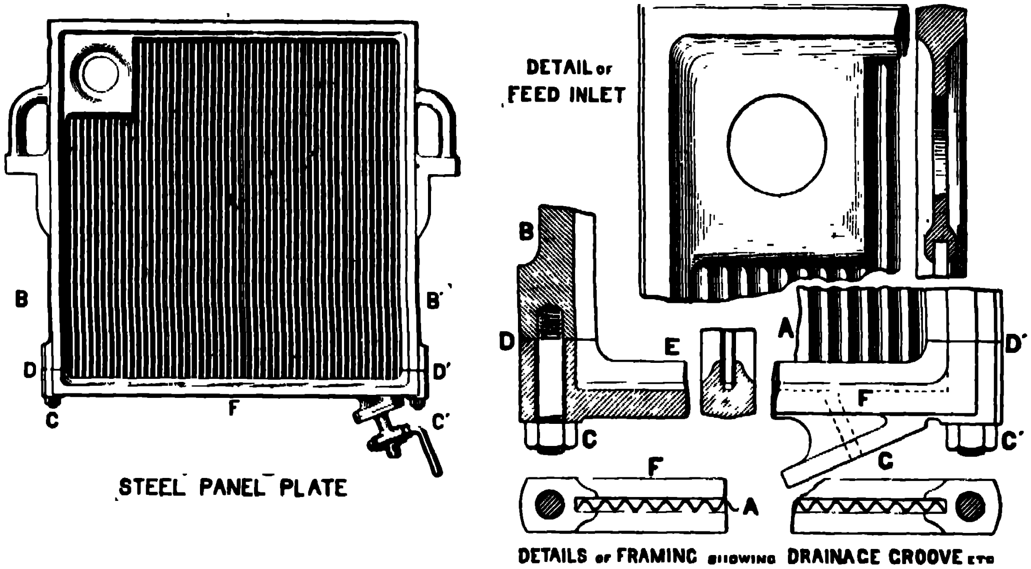


FIG. 37 —Construction of Steel Panel Plate.

Bleaching with Fullers' Earth.—The bleaching-tank is a cylindrical sheet-steel tank and is provided with a closed steam-coil for maintaining the requisite temperature, which need not exceed 125° Fahr., and with either an air-blast or a mechanical agitator for effecting intimate admixture of the stock and earth. The proportion of fullers' earth used depends upon the quality of the stock, and as a rule does not exceed 1 to 2 per cent. It is sifted upon the surface of the liquid stock and the whole is well mixed. The progress in the absorption of impurities by the earth may be observed from time to time by filtering a sample of the mixture through filter-paper into a 4-ounce oil-sample bottle and comparing the color of the filtered stock with a previous sample. When the color of two successive samples thus treated is uniform the maximum bleaching effect may be assumed to be obtained. With the mixture of stock and earth in agitation the whole is pumped through the filter-press, the filtrate therefrom being conducted to storage-tanks or to the soap-kettle, as desired.

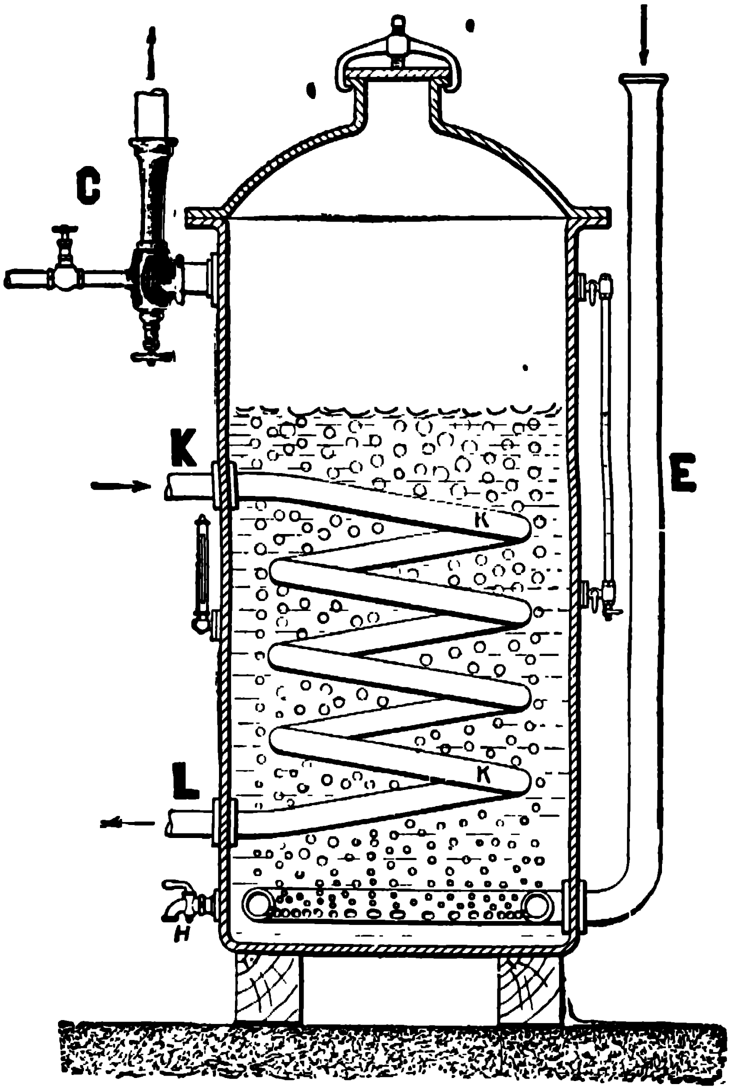


FIG. 38.—Apparatus for Bleaching Palm-oil.

Press-cake.—The residue left in the filter-press and forming the cake is fullers' earth plus absorbed organic matter, with more or less retained stock. The quantity of the latter may be reduced to a minimum by steaming out the press. The disposition of the stock thus separated by steam will be determined by its quantity and quality. The stock yet remaining in the cake, while of considerable amount in the long run, is valueless considering the expense of its recovery. The press-cake is a worthless by-product.

The following analyses of press-cake obtained in bleaching grease will serve to indicate its general characteristics:

TABLE XVI.—ANALYSES OF PRESS-CAKE.

Water Per Cent.	Stock. Per Cent.	Earth Per Cent.	Impurities from Stock Per Cent
7.29	24.33	51.52	16.80
7.20	14.19	49.14	29.17
4.08	21.35	55.29	15.38
4.12	23.28	44.55	28.05
1.22	21.34	52.77	18.67
2.02	22.16	55.29	19.63

Bleaching of Palm-oil by Oxygen of the Air.—A device for the bleaching of palm-oil by means of the oxygen of the air is shown in Fig. 38. The bleaching apparatus consists of a closed iron vessel, on the upper part of which is fixed a steam-jet exhauster, marked *C*. In the vessel is a copper heating coil, *K*, and at the bottom an annular air-distributing pipe with a large number of small holes, connected to an air inlet pipe, *E*, carried outside the vessel above the level of the oil. The palm-oil is first liquefied in a tank, and is then allowed to stand for a few hours, so that the water and dirt may settle to the bottom.

The liquid oil is then poured into the bleaching-vessel, the top cover is closed, and steam is admitted to the copper coil, until the oil is raised to a temperature of 212° Fahr. The exhauster is then started and air drawn through pipe *E*, and through the oil.

The air issuing through the small holes of the distributing-pipe comes into intimate contact with the oil, and the oxygen of the air is thus fully utilized for bleaching.

As no acids or chemicals are used in this process, noxious gases are not formed, and the natural properties of the oil are in no way affected. The discharge-pipe from the exhauster should be carried through the roof, so that all steam from the exhauster and vapor from the oil is discharged outside.

The process of producing a good oil takes about two hours, and it is only at the beginning that attention need be paid to the apparatus.

For the rest of the time no attention is necessary. The bleaching-vessel may vary in capacity from 25 to 500 gallons.

CHAPTER V.

THE CHEMICAL CHARACTERISTICS OF SOAP-STOCK AND THEIR BEHAVIOR TOWARDS SAPONIFYING AGENTS.

Classification of Soap-stock. Class I. Cocoanut- and Palm-kernel Oils. Class II. Olive-, Corn-, and Cottonseed-oils. Class III. Tallow and Palm Oil. Class IV. Linseed- and Castor-oils. Class V. Red Oil and Rosin. Soap-makers' Percentage. Saponifying Agents.

Classification of Soap-stock.—In the preceding pages we have considered the source and preparation of the chief raw materials of soap manufacture. The different organic bodies display different and characteristic properties in the process of combination with alkali. As this difference resides in the chemical composition of the bodies, it is possible to classify soap-stock in accordance with their chemical composition and behavior towards saponifying agents.

TABLE XVI(*a*).—CLASSIFICATION OF SOAP-STOCK.

I.	II.	III.	IV.	V.
Cocoanut-oil Palm-kernel oil	Olive-oil Olive-oil—Foots Corn-oil Cottonseed-oil Sunflower-seed oil Peanut-oil	Tallow Palm-oil Greases	Linseed-oil Castor-oil	Red oil Rosin

As has already been explained the combination of a fat or oil with caustic alkali is a progressive and stepwise reaction based upon the constitution of the former bodies as triglycerides. It may be noted in this connection that the reactions of organic bodies do not proceed with the same ease and certainty that characterize the reactions of inorganic bodies. Combination in the majority of cases does not ensue immediately, although the reacting bodies may be in intimate contact, and in almost every case especial pains must be taken to complete the reaction, as is done in the strengthening change of boiled soaps.

Class I. Cocoanut- and Palm-kernel Oils.—These oils stand alone with respect to the large quantity of alkali absorbed, the high density of the alkali solutions required for saponification, the firmness and high yield of the resulting soap and its capacity for the absorption of water and mineral filling matter, and the large quantity of salt required for graining, aside from certain peculiarities of the stock which particularly adapt it for soap-manufacture by the cold process. These characteristics are explained by the composition of the oils themselves. Two samples of commercial cocoanut-oil, examined by Ulzer,* analyzed about 2.32 per cent of the glycerides of volatile fatty acids (chiefly caproic and caprylic acids), and about 10.45 per cent of olein, the remainder consisting of laurin and myristin with a little caprin. The presence of palmitin is regarded as doubtful. Oudemans gives the following as the composition of palm-kernel oil: olein, 26.6 per cent; stearin, palmitin, myristin, 33 per cent; laurin, caprin, caprylin, caproin, 44.4 per cent. Although palm-kernel oil produces a soap of essentially the same character as that made from cocoanut-oil, the meagre source of the supply in comparison with that of cocoanut-oil greatly restricts its use.

Cocoanut-oil saponifies readily with concentrated lye and saponification, when once started, proceeds with great rapidity. Much heat is evolved in the process of combination. With weak lye, 12° Bé., which density is most suitable for tallow saponification, combination does not begin until the lye has been concentrated by the evaporation of the superfluous water. The soap formed is thin, very fluid and lacks the characteristic body of a tallow soap. During saponification by the boiled processes, cocoanut-oil swells considerably and it is best, in order to avoid running over the kettle and to more satisfactorily work the contents, to saponify the total charge in successive portions. Frequently saponification may ensue so rapidly that the entire contents of the kettle will "set" to a firm mass. To avoid the troublesome after-manipulation thus made necessary, it is best to retard saponification, when such a tendency appears, by the addition of dry salt, or brine, and to vigorously work the mass through with live steam.

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* Chem. Rev. Fatt- und Harz Ind., 6 [11], 203, 204.

Pure cocoanut-oil soap possesses a peculiarly caustic taste, even though no free alkali be present. Owing to the solubility in water and dilute brine of the alkali salts of the fatty acids of cocoanut-oil, a much greater quantity of salt is required for graining than with animal and other vegetable soap-stock. Owing to the fluidity of the soap and its capacity for water and to obtain economy of kettle-space, the use of dry salt in graining is preferable. The waste lye from a well-grained cocoanut-oil tallow soap, containing 50 per cent of the former ingredient, will contain almost twice as much salt as a waste lye from a tallow cottonseed-oil soap. Thorough graining of settled cocoanut-oil soaps is necessary to prevent the soap being retained in solution in the hot waste lye, which separates as a gelatinous mass of soft soap, on the cooling of the latter.

It is advisable to hold waste lye from cocoanut-oil soap in storage until chilled, to allow the return of the separated soap to the kettle.

Cocoanut-oil soap is white, brittle, and hard. It lathers freely in both fresh and salt water. The lather is formed quickly, is of thin body and lacks permanency. In the manufacture of settled soap cocoanut-oil is a valuable addition to tallow and cottonseed-oil, the product of such a mixture being superior to that produced from either stock alone. Cocoanut-oil soaps almost invariably become rancid with age and betray the presence of the oil in the offensive odor. This may be overcome to a degree by perfect saponification. The natural compactness of cocoanut-oil soap lends itself, in mixed stock, to overcome the "cracking" to which all settled soap is susceptible. Cocoanut-oil of good quality for soap-makers' use is considered to liberate in the kettle about 12 per cent of glycerin.

Cocoanut and palm-kernel oils liberate more glycerin than any other oil used in soap making, which is in harmony with their high absorption of alkali.

Nagel made numerous experiments for the purpose of finding out why cocoanut-oil is easily saponified in the cold way, all other fats and oils used in the soap industry needing considerable heat for saponification. In the first place it was necessary above all to look at the chemical composition of these fats, all other fats being composed of the glycerides of the higher fatty acids, such as stearic, oleic, and palmitic acid. Cocoanut-oil is composed of much lower fatty acids, chiefly myristic, caproic, caprylic and capric acids. But from this alone the cold saponification cannot be understood, because there is no great difference in the saponification of the glycerides of myristic and stearic acids. Now it is known that the glyceride of myristic acid, by the influence of air and moisture, is easily decomposed (this process causing the state called rancidity), whereby lower fatty acids, lactones, and other products are obtained, which are easily saponified. Cocoanut-oil so quickly becomes rancid that in the market only the rancid product can be bought, which is used for soap-making.

It is now well known that the glycerides of the lowest fatty acids can be easily saponified in the cold way, yielding great heat.

This property decreases with the decreasing solubility in water and with the increasing number of carbon atoms in the molecule, so that the glycerides of the higher fatty acids can only be saponified under comparatively great heat.

Nagel further states that if the saponification has taken place, the same continues also to fats which are not easily saponified at a far lower than the usual temperature. This is effected by the kalalytic action of the ions of hydrogen and goes on in a logarithmic sequence.

If we have now a mixture of fats which saponify with difficulty with fats which are easily saponified, the saponification takes place at a temperature far lower than if the latter were present.

The saponification which begins at a low temperature by the presence of other low fatty acids, continues and induces the further saponification of the higher fatty acids.

In this simple way also the cold saponification of cocoanut-oil, which contains always quantities of the lower fatty acids and other substances accompanying rancidity, which are easily saponified, can be explained. These substances begin the saponification which continues with the higher fatty acids. A similar process takes place in saponifying rancid butter and other rancid fats, i.e., fats containing the lower fatty acids and substances which are easily saponified.

Class II. Olive-, Corn-, and Cottonseed-oils.—Just as the fatty acids of glycerides of low molecular weight are characteristic, and give rise to the peculiarities of the soap-stock of the preceding class, the glyceride olein is characteristic of the soap-stock of this class and imparts to it its properties.

Olive-oil is the representative non-drying oil. It saponifies readily with caustic lyes of variable densities, producing a light-colored to greenish soap, according to the quality of the oil and yields readily a thin, slimy lather peculiar to all soaps made from stock in which olein predominates. The color of olive-oil and olive-oil foots soaps gradually disappears on ageing. Olive-oil produces a mildly cleansing soap for toilet purposes and, being free from the unsaturated glycerides of the drying oils, is an ideal stock for textile soap.

The remaining oils of this class are semi-drying oils, of which sunflower-seed and peanut-oils are of no importance in the United States as soap-stock. Cottonseed and corn-oils closely resemble each other in their chemical characteristics, and in their behavior towards saponifying agents, are practically identical. Refined cottonseed-oil saponifies with difficulty, especially when used without admixture of animal stock, and only after long-continued boiling. Saponification is best begun with caustic lye of 15° Bé. It ensues more readily with the crude oil owing to the presence of free fatty acids and may be hastened with the refined oil by addition of soap-scrap or admixture with easily saponifiable fats. Unless carefully refined and thoroughly saponified, soap made from cottonseed-oil is subject to rapid deterioration in appearance through discoloration and rancidity. The chief disadvantage of cocoanut and cottonseed-oils is their proneness to rancidity. Cottonseed-oil soap is not so easily grained as tallow. It is naturally of thin body, which appearance should not be confused with excess of water. Fresh cottonseed-oil soap is white, but discolors rapidly.

Cottonseed-oil is the chief commercial vehicle for the glyceride olein. It is employed, as a rule, in admixture with varying proportions of tallow and grease. A marketable settled soap of cottonseed-oil is not practicable. Soap thus made is "thin" and lacks the body of a tallow soap. By the use, however, of a large proportion of soda-ash and sodium silicate in crutching, an artificial firmness may be imparted. The only household soap on the market having an exclusively cottonseed-oil origin is that made of cottonseed-oil soap-stock. The use of this material in the manufacture of domestic purpose soap is rapidly extending, and in many parts of the South is sold almost to the exclusion of tallow laundry-soap. Thorough elimination of the albuminous and muscilaginous matter and as complete a separation of the coloring-matter as is possible from the crude stock, with generous application of soda-ash and sodium silicate, admit of a surprisingly fine piece of laundry soap, apparently well adapted to an uncritical taste and the prevailing hard water of the South.

Sodium oleate is soluble in ten parts of water, while sodium stearate we have seen is not appreciably affected by this volume. In accordance with its greater solubility we note its peculiar lathering properties. It lathers more readily than sodium stearate, but instead of the firm lather of tallow soap obtained only after much rubbing, we have a slimy lather peculiar to all soaps made of oils consisting largely of olein, viz., castile and cotton-oil soap-stock.

The great affinity of sodium oleate for water manifests itself in the readiness with which soaps containing a large proportion of it "sweat." This tendency at its maximum is met with in unfilled soap made from cottonseed-oil soap-stock. This soap in entirely sodium oleate. So marked is this tendency that any considerable increase in atmospheric humidity is shown by the accumulation of a film of moisture upon the surface of the soap. Cottonseed-oil will liberate about 10 per cent of glycerin in the kettle.

Class III. Tallow.—The characteristic glyceride of soap-stock of this class is stearin and palmitin with variable proportions of olein. Tallow in its various grades and palm-oil admit of ready saponification with weak caustic lye, preferably 10° to 12° Bé., and more so as the percentage of free fatty acids increases, as in greases and the lower grades of palm-oil. Soap-stock of this class makes a firm soap with good body, readily grained, and in its behavior in the kettle gives well-defined indications of the progress of saponification. Weak caustic lye is absolutely necessary for the saponification of neutral stock of this class, otherwise the first-formed soap is immediately thrown from solution and the process of saponification retarded. With concentrated caustic lye the fat will float upon the surface and emulsification will not begin until the former has been diluted either by water from condensed steam or that added for the direct purpose of dilution.

It is common practice in the boiling of settled soap to make, as a rule, lye of high concentration and to dilute it as required, usually as the caustic lye is added to the kettle. By this method smaller caustic tanks are required with resulting economy of space.

As shown in Table III, tallow gives the least yield of anhydrous soap of all the glyceride stock commonly employed. It requires the least quantity of salt for graining, being sufficiently grained with from 8 to 10 per cent of salt in the waste lye. It produces a white soap according to the quality of the stock.

Sodium stearate, which constitutes the bulk of the dry soap, is one of the least soluble in water of all the alkaline salts of the fatty acids, it undergoing practically no change when treated with ten parts of water; neither is its hardness appreciably affected. This quality manifests itself in the inferior lathering properties of pure tallow soap. Furthermore, tallow soap on aging becomes so hard as to be used only with more or less injury to the fabric. Its comparative insolubility in water manifests itself in the readiness with which it dries. Its presence in admixture with other fats imparts firmness or body to the soap, thus enabling a greater proportion of softer stock, or that fatty acid whose sodium salt has a greater affinity for water to be incorporated. Its use thus increases the amount of water that may be added to a soap without an excessive sacrifice of firmness. Sodium palmitate so closely resembles sodium stearate that the statements concerning the influence of the latter salt are directly applicable. Palm-oil, of which palmitin is the chief glyceride, is of limited use in the manufacture of American laundry soaps. It is employed to some extent to disguise the odor of rosin in excessively rosined soaps. In Great Britain and Continental Europe it replaces tallow to a large degree.

The coloring-matter of palm-oil is not destroyed by saponification, but persists in the soap made from it. It is destroyed, however, by chemical bleaching processes, the bichromate process originally proposed by Watts being usually employed, whereby a fine light-colored stock is obtained approaching the consistency of grease. Palm-oil is used to some extent as a base for textile soap and for this purpose stands next to olive-oil. Stock of this class, particularly tallow, is the standard soap-stock and for boiled soaps is the basis of the blending with other fats and oils. The various fats and oils in admixture in the process of saponification tend to impart their properties to each other. Stock of Class I requires lye of high density for saponification, not less than 20° Bé.; stock of Class II and Class III is satisfactorily worked with lye so dilute, 12–15° Bé., as to be unsatisfactory with stock of Class I. When these various fats and oils are mixed, as is frequently done in the manufacture of laundry soap and toilet-soap base, the requirements of each are modified so that a stock requiring for saponification alone a concentrated lye combines with lye of a density with which it would not otherwise combine without troublesome manipulation. On the other hand, a stock saponifying alone readily with weak lye, combines, in admixture with stock of high alkali absorption, with more concentrated lye. The same principle holds in the formation of lather, which is a prime consideration in the manufacture of soap, either settled or milled, for toilet purposes. Soap made from stock of Class I yields a quick, profuse but thin lather; from Class II an abundant slimy lather; from Class III, a slow, thick but lasting lather. Tallow and good grease will liberate in the kettle about 9 per cent of glycerin.

Class IV. Linseed- and Castor-oils.—Linseed- and castor-oils are of very limited use in the United States as soap-stock, being employed exclusively for special soaps, as the *Sapo viridis*, or green soap, of the pharmacy and transparent soap. Stock of this class is very readily saponified and yields a quick-lathering, very soluble soap. Linseed-oil is used to a great extent in European countries for soft soap. The soap is of pale-brown color and of characteristic odor.

Class V. Red Oil and Rosin.—As seen in Chap. III, red oil is commercial oleic acid, and in its behavior toward saponifying agents may be classed with rosin, which is essentially an acid body. As no glycerin is to be liberated, there is no saponification, combination consisting of direct saturation with alkali. It is possible to effect combination either in whole or part with soda-ash, which is much cheaper than caustic soda, but the practical disadvantages outweigh the apparent economy. Red-oil or "oleine" soap is used to a large extent in the textile industry and will be discussed under Textile Soaps. Consisting chiefly of oleic acid, soap made from red oil possesses the general properties common to soap made from stock of Class II.

Rosin "soap" is a soft, sticky body of dark-brown color, according to the quality of the rosin. Consisting when in a pure state entirely of organic acids, rosin permits of saturation with either caustic or carbonated alkali. It is rarely "killed" alone or by means of soda-ash, but with caustic soda, and usually after the glyceride stock has been added to the kettle and killed.

The alkaline salts of the rosin acids are very hygroscopic, naturally readily soluble in water, and make a very soft soap. They detract from the firmness of all soaps in which they enter. Their presence is betrayed by a stickiness due to the marked affinity of the alkaline resinates for moisture, which peculiarity manifests itself in the readiness with which rosined soaps "sweat." Their detergent properties, ready solubility, and cheapness make them an indispensable addition to the firmer tallow, whose hardness they greatly ameliorate. The amount of rosin that a fat or oil will assimilate without becoming unduly soft depends upon the percentage of stearin in the fat or oil. A good firm tallow will carry much more rosin than a soft grease and the relative proportions of fat and rosin to use are determined by the grade of soap it is desired to make. The presence of rosin in a soap invariably is betrayed by its stickiness, and to reduce this "sweating" to a minimum with a constant percentage of rosin, the quality of the fat-stock must be improved. A good grease will carry 25 to 50 per cent of rosin without the soap becoming excessively soft. Soap containing rosin becomes darker with age, while a soap made from unbleached palm-oil retains its yellow color even when rosin is used, although without the presence of rosin the characteristic color of the palm-oil would gradually disappear on exposure to light and air.

Soap-makers' Percentage.—By soap-makers' percentage is understood a method of expressing the proportions of glyceride stock and rosin with which the kettle is charged. A 100-per cent rosin soap contains equal parts of fat and rosin; a 50-per cent rosin soap contains 3 parts fat and 1 part rosin; a 150-per cent rosin soap contains the 1 part fat and 3 parts rosin.

A more rational method to express the proportions is the percentage rosin on the basis of 100 pounds of fat-stock. Thus a mixture of 75 parts fat and 25 parts rosin by soap-maker's percentage is called a 50-per cent rosin soap. This is equivalent to 100 pounds of fat-stock and 33⅓ pounds of rosin or 33⅓ per cent of rosin on the basis of 100 pounds of fat-stock.

Saponifying Agents. Caustic Soda and Caustic Potash.—The greater cheapness of caustic soda restricts the use of caustic potash to soaps of special character. Although caustic potash is the more energetic base, in fact the most strongly basic of all the metals, potash-soaps are notable over soda-soaps for their mildly detersive and less harmful action both upon the skin and upon animal and vegetable fibres. The more active properties of the alkali are transferred to the soap made with it, hence a potash or a mixed soda and potash-soap is more soluble than a soda-soap and, as a result, is more active as a detersive agent and less soap is therefore required; it is of tougher texture and owing to the affinity of potash-salts for moisture, it is characterized by the soft consistency. In shaving-soap potash is a necessary and essential ingredient. Potash, being of higher molecular weight (56), will show a greater yield in soft soap than caustic soda, whose molecular weight is 40. The definition of soft soap has been—one made with potash. This definition is not strictly accurate. A soft soap in modern practice is determined by the degree of hydration and not alone by the character of the alkali used. It is possible to prepare a soap of a fair degree of firmness with potash.

In the cold process especially, a proportion of potash causes a marked improvement in the product. It renders the soap milder, more translucent at the edges and in every respect a better article in use and appearance.

Saponification with Soda-ash.—Soda-ash is a salt, i.e., a compound of an acid, viz., carbonic acid, and a base, viz., caustic soda, and while possessing alkaline properties its chemical activity is too low to admit of combination with neutral glycerides. It combines with fatty acids and rosin, which is an acid body, with the liberation of carbonic acid gas, and is used to some extent for the saturation of these bodies. Its use for this purpose in connection with red oil and rosin is described at length on subsequent pages.

CHAPTER VI.

MECHANICAL EQUIPMENT OF THE SOAP FACTORY.

General Construction and Location of Factory. Receipt and Handling of Raw Materials. Caustic Melting- and Storage-tanks Grinding Caustic Soda. The Soap-kettle. The Rotary Pump. The Strainer. Filler Mixer. The Crutcher. Use of Crutcher. The Frame. The Slabber. The Cutting-table. The Remelter. Drying of Soap. The Drying-room. Installation of Disc-fan. The Foot-press. Pressing Soap. Soap Dies. The Power-press. Soap Wrapping. The Soap-box. Summary of Factory Equipment.

General Construction and Location of Factory.—The course of manufacture of any commodity should pursue a straight line with the least labor involved in its transportation; when elevated, its descent should be facilitated by gravity and with course of manufacture along vertical or horizontal lines, or a combination of both, its course should not be retraced but should pursue a definite, progressive movement as its manipulation approaches completion and it is shipped from the factory. With high land-value, requiring the erection of tall buildings, this course, as a rule, is upward with the raw material and downward as the product approaches completion. With converse conditions the course of manufacture is in general more along horizontal lines. As the industry has developed, building construction has greatly improved until, under the most improved conditions, the factory structure is fire-proof, with cement floors, capacious elevators, machinery electrically operated from a central power plant, which provides as well ample steam supply for soap-boiling, waste-lye evaporation and glycerin distillation. As in all lines of industry, various degrees of manufacturing efficiency naturally prevail. Location is an important consideration; wherever practicable, the factory should be located with immediate access to tidewater or railroad, or both, for easy receipt of raw material and shipment of the manufactured product. A yard of convenient size

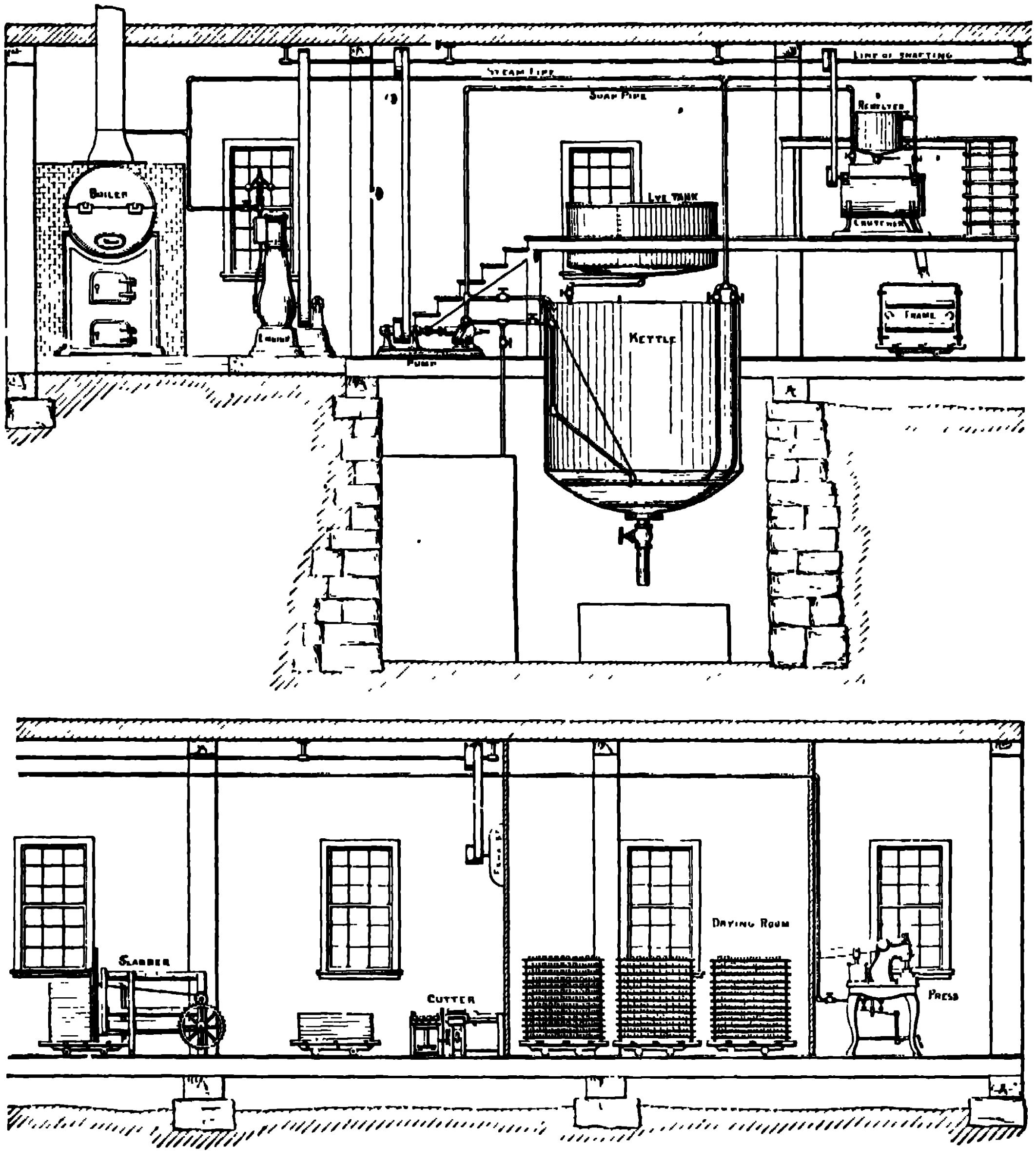


Fig. 39.—Conventional View of Soap Manufactory Showing Essential Equipment and Course (Left to Right) of Procedure.

is almost indispensable for the storage of rough material. In Fig. 39 is shown in its simplest form the mechanical equipment essential to soap manufacture, the course of manufacture being from left to right.

Receipt and Handling of Raw Material.—Glyceride stock, in its various grades, is received at the factory in two styles of containers, viz., barrels and tank-cars. Wherever the volume of business and location of the factory will warrant, the receipt of the tallow in tank-cars represents the maximum in cleanliness and convenience that at present seems to be possible. Tank-cars will hold, as a rule, about 40,000 pounds of stock, equivalent to over 100 barrels, which surpasses the capacity of a box-car. When one considers that with suitable piping, pumps, scale, and storage-tanks, a tank-car can be emptied in about an hour, the superiority of this method of receiving tallow over that involving the use of steam in emptying barrels, requires no further comment.

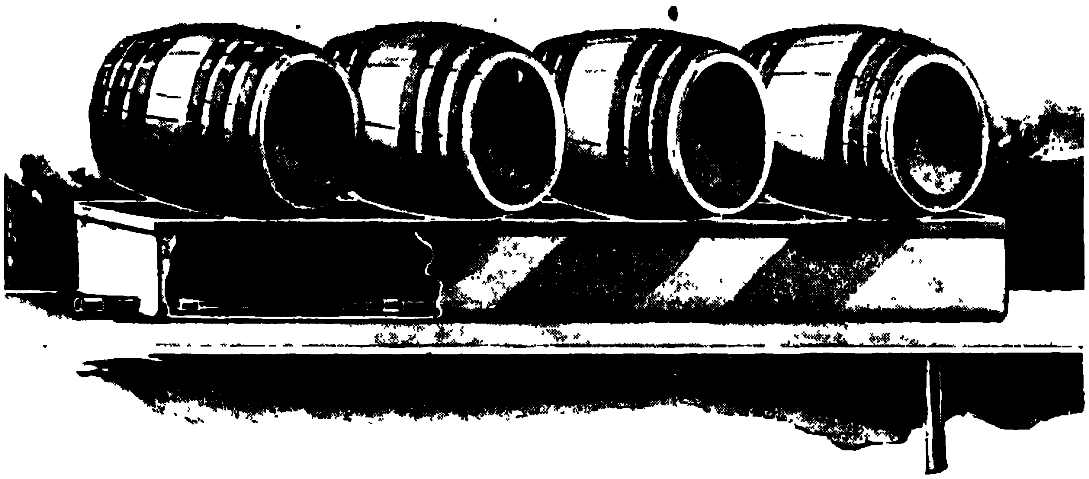


FIG. 40.—Stock-steamer.

Rosin is received in rough barrels which may be broken up at once and the rosin used, or stored in the yard until required. Silicate of soda is received in either iron drums or barrels and in the latter case should be protected from the weather. It may be used either directly from the barrel or, when used in large amount, it is more convenient to transfer it to a tank from which it may flow by gravity. It may also be received by tank-car. Caustic soda is received in thin sheet iron drums and may be stored in the yard. It may also be received in concentrated solution in tank-cars. Soda-ash is received in bags of 250–300 pounds. Glyceride stock, received in barrels, is emptied from the container by the injection of free steam into the barrel, which is placed in a shallow sheet-iron trough from which the stock may flow by gravity either directly into the kettle or storage-tank. The principle of the stock-steamer is shown in Fig. 40.

The stock-steamer should be in a closed compartment to prevent the egress of odor and vapor, and may be of any size, according to the rapidity with which it is desired to empty a given amount of stock.

Caustic Melting- and Storage-tanks.—Tanks for caustic lye may be of circular or rectangular cross-section, should be well riveted and caulked, and of a capacity determined by the amount of stock killed in a given time. The tank should be located so that the contents may flow by gravity to the kettle. By the most convenient arrangement one tank suffices for both melting the caustic and storing the caustic lye. A pipe should be provided for the introduction of water and an outlet at least 3 inches from the bottom for the discharge of the strong lye. A water-pipe should be connected with the strong lye pipe by means of a T and the flow of both liquids regulated by means of conveniently located valves, whereby the strong caustic lye may be diluted in the pipe leading to the kettle to any desired density. The caustic-lye tank should contain a grating, either supported from the bottom or suspended from the sides of the tank and preferably inclined towards one end, whereby the cylinders of solid caustic with the sheet-iron covering removed may be introduced at one end and roll to the opposite. With the desired number of drums added in this manner, water is added and solution of the caustic suspended in the upper portion of the water begins. A circulation of light and heavy liquor is immediately induced, with the result that a solution of any desired

TABLE XVII.—CAPACITIES AND DIMENSIONS OF CYLINDRICAL TANKS.

Capacity, gals.	60	80	100	120	140	160	180	200	300	400	500	600	700
Diameter, ins.	30	30	36	36	36	36	39	39	48	48	60	60	60
Depth, ins.	20	27	24	28	32	38	36	40	40	52	42	50	58

Capacity, gals.	800	900	1000	1200	1400	1600	1800	2000	2500	3000	3500	4000
Diameter, ins.	66	66	69	75	75	84	84	90	96	96	108	108
Depth, ins.	55	61	62	65	75	68	76	73	80	96	89	101

density is soon produced according to the number of drums of caustic added. So long as solid caustic remains water may be added to replace the heavy liquor withdrawn.

Grinding Caustic Soda.—As the preparation of ground caustic soda is an industry associated with soap manufacture an improved device to effect the reduction of the hard commercial caustic soda to a size suitable for packing in cans is shown in Fig. 41. In its mechanical construction the device is of the nature of a soap-chipping machine, and for its purpose is superior to other methods of pulverizing caustic soda. The knives of the grinder are detachable and have serrated edges and are made of material especially adapted to the severe usage to which they are subjected. The caustic soda in drums may be broken up into fragments of suitable size, either in the closed compartment where the grinder is located or on the floor above, from whence the grinder may be charged by gravity. The pulverized caustic soda may be collected in a suitable receptacle on the

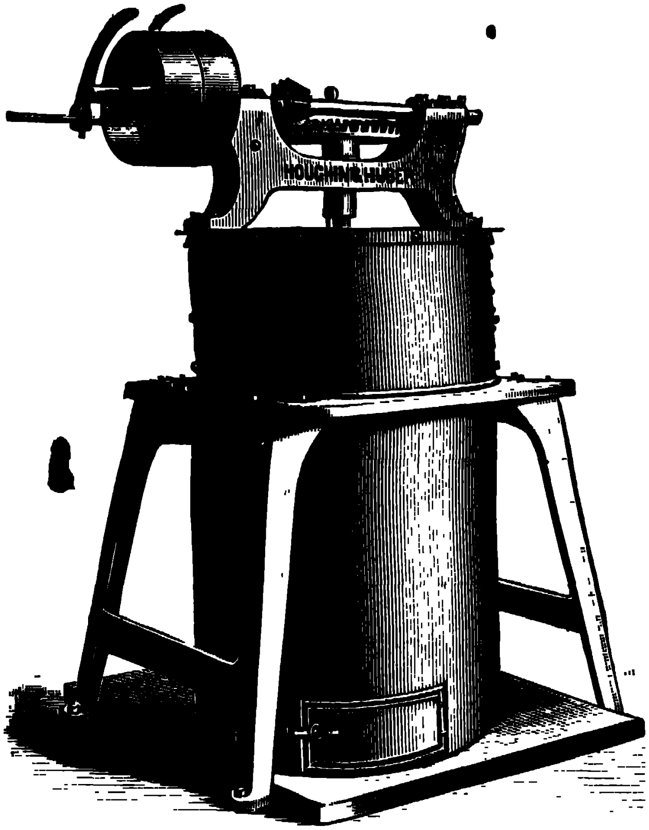


FIG. 41.—Caustic-soda Grinder.

TABLE XVIII.—SPECIFIC GRAVITIES OF SOLUTIONS OF CAUSTIC SODA. (HURST.)

Degrees Twaddell.	Degrees Beaumé	Specific Gravity.	Per Cent by Weight of		Lbs. of actual NaOH contained in 1 gallon of lye made from commercial caustic of		
			Na_2O.	NaOH.	77 Per Ct.	74 Per Ct.	70 Per Ct.
1	0.7	1.005	0.368	0.474	0.048	0.046	0.043
2	1.4	1.010	0.742	0.957	0.097	0.092	0.087
3	2.1	1.015	1.114	1.436	0.146	0.131	0.129
4	2.7	1.020	1.480	1.909	0.194	0.185	0.180
5	3.4	1.025	1.834	2.365	0.243	0.231	0.219
6	4.1	1.030	2.194	2.830	0.291	0.278	0.262
7	4.7	1.035	2.521	3.252	0.335	0.320	0.303
8	5.4	1.040	2.964	3.746	0.389	0.371	0.350
9	6.0	1.045	3.244	4.184	0.438	0.417	0.393
10	6.7	1.050	3.590	4.631	0.486	0.461	0.438
11	7.4	1.055	3.943	5.086	0.536	0.510	0.483
12	8.0	1.060	4.292	5.536	0.586	0.558	0.528
13	8.7	1.065	4.638	5.982	0.636	0.607	0.573
14	9.4	1.070	4.972	6.413	0.680	0.653	0.617
15	10.0	1.075	5.311	6.911	0.742	0.707	0.668
16	10.6	1.080	5.648	7.285	0.786	0.749	0.709
17	11.2	1.085	5.981	7.715	0.836	0.798	0.755
18	11.9	1.090	6.317	8.140	0.886	0.845	0.800
19	12.4	1.095	6.639	8.564	0.937	0.894	0.846
20	13.0	1.100	6.954	8.970	0.986	0.941	0.890
21	13.6	1.105	7.276	9.386	1.037	0.989	0.938
22	14.2	1.110	7.594	9.796	1.087	1.037	0.981
23	14.9	1.115	7.910	10.203	1.137	1.123	1.026
24	15.4	1.120	8.223	10.607	1.187	1.175	1.071
25	16.0	1.125	8.533	11.107	1.238	1.181	1.117
26	16.5	1.130	8.893	11.471	1.296	1.237	1.170
27	17.1	1.135	9.251	11.933	1.354	1.292	1.222
28	17.7	1.140	9.614	12.401	1.413	1.350	1.277
29	18.3	1.145	9.965	12.844	1.470	1.413	1.337
30	18.8	1.150	10.313	13.303	1.529	1.460	1.381
31	19.3	1.155	10.666	13.859	1.600	1.528	1.445
32	19.8	1.160	11.008	14.190	1.646	1.541	1.456
33	20.3	1.165	11.347	14.637	1.705	1.627	1.539
34	20.9	1.170	11.691	15.081	1.764	1.684	1.593
35	21.4	1.175	12.025	15.512	1.822	1.739	1.645
36	22.0	1.180	12.356	16.139	1.904	1.817	1.719
37	22.5	1.185	12.692	16.372	1.942	1.853	1.753
38	23.0	1.190	13.016	16.794	1.998	1.887	1.804
39	23.5	1.195	13.339	17.203	2.055	1.962	1.856
40	24.0	1.200	13.660	17.620	2.122	2.026	1.916
41	24.5	1.205	14.058	18.133	2.185	2.085	1.973
42	25.0	1.210	14.438	18.618	2.252	2.147	2.033
43	25.5	1.215	14.823	19.121	2.323	2.221	2.097
44	26.0	1.220	15.124	19.613	2.392	2.280	2.161
45	26.4	1.225	15.502	19.997	2.444	2.338	2.206
46	26.9	1.230	15.959	20.586	2.562	2.417	2.285
47	27.4	1.235	16.299	20.996	2.593	2.475	2.341
48	27.9	1.240	16.692	21.532	2.669	2.548	2.410
49	28.4	1.245	17.060	22.008	2.739	2.615	2.474
50	28.8	1.250	17.424	22.476	2.809	2.681	2.536

TABLE XVIII.—SPECIFIC GRAVITIES OF SOLUTIONS OF CAUSTIC SODA (*Continued*)

Degrees Twaddell.	Degrees Beaume.	Specific Gravity.	Per Cent by Weight of		Lbs. of actual NaOH contained i 1 gallon of lye made from commercial caustic of		
			Na_2O.	NaOH.	77 Per Ct.	74 Per Ct.	70 Per Ct
51	29.3	1.255	17.800	22.962	2.881	2.750	2.602
52	29.7	1.260	18.166	23.433	2.952	2.818	2.666
53	30.2	1.265	18.529	23.901	3.020	2.886	2.730
54	30.6	1.270	18.897	24.376	3.095	2.955	2.795
55	31.1	1.275	19.255	24.858	3.171	3.027	2.863
56	31.5	1.280	19.609	25.295	3.237	3.090	2.932
57	32.0	1.285	19.961	25.750	3.308	3.158	2.988
58	32.4	1.290	20.318	26.210	3.381	3.227	3.053
59	32.8	1.295	20.655	26.658	3.452	3.364	3.117
60	33.3	1.300	21.156	27.110	3.524	3.394	3.182
61	33.7	1.305	21.405	27.611	3.603	3.439	3.253
62	34.2	1.310	21.785	28.105	3.682	3.514	3.224
63	34.6	1.315	22.168	28.595	3.760	3.593	3.395
64	35.0	1.320	22.556	29.161	3.849	3.674	3.475
65	35.4	1.325	22.926	29.574	3.919	3.712	3.539
66	35.8	1.330	23.310	30.058	3.997	3.816	3.610
67	36.2	1.335	23.670	30.535	4.072	3.891	3.681
68	36.6	1.340	24.046	31.018	4.156	3.967	3.754
69	37.0	1.345	24.410	31.490	4.232	4.042	3.824
70	37.4	1.350	24.765	31.948	4.312	4.116	3.894
71	37.8	1.355	25.152	32.446	4.396	4.196	3.970
72	38.2	1.360	25.526	32.930	4.478	4.274	4.043
73	38.6	1.365	25.901	33.415	4.561	4.354	4.109
74	39.0	1.370	26.285	33.905	4.645	4.434	4.194
75	39.4	1.375	26.650	34.382	4.728	4.513	4.269
76	39.8	1.380	27.021	34.855	4.810	4.592	4.344
77	40.1	1.385	27.385	35.328	4.893	4.670	4.418
78	40.5	1.390	27.745	35.795	4.975	4.794	4.493
79	40.8	1.395	28.110	36.258	5.058	4.828	4.567
80	41.2	1.400	28.465	36.720	5.141	4.907	4.642
81	41.6	1.405	28.836	37.203	5.227	4.989	4.720
82	42.0	1.410	29.203	37.674	5.312	5.071	4.797
83	42.3	1.415	29.570	38.146	5.397	5.135	4.873
84	42.7	1.420	29.930	38.610	5.482	5.233	4.950
85	43.1	1.425	30.285	39.071	5.567	5.314	5.027
86	43.4	1.430	30.645	39.530	5.653	5.396	5.104
87	43.8	1.435	30.995	39.986	5.738	5.467	5.181
88	44.1	1.440	31.349	40.435	5.823	5.558	5.258
89	44.4	1.445	31.700	40.882	5.908	5.640	5.335
90	44.8	1.450	32.043	41.335	5.923	5.721	5.412
91	45.1	1.455	32.460	41.875	6.093	5.816	5.502
92	45.4	1.460	32.870	42.400	6.191	5.909	5.608
93	45.8	1.465	33.283	42.935	6.290	6.004	5.679
94	46.1	1.470	33.695	43.467	6.389	6.009	5.769
95	46.4	1.475	34.092	43.980	6.487	6.193	5.856
96	46.8	1.480	34.500	44.505	6.586	6.287	5.948
97	47.1	1.485	34.899	45.013	6.685	6.381	6.035
98	47.4	1.490	35.245	45.530	6.784	6.476	6.126
99	47.8	1.495	35.691	46.041	6.884	6.571	6.216
100	48.1	1.500	36.081	46.545	6.982	6.665	6.303

floor below the one on which the grinder is situated, or it may be removed from the grinder through a door provided for that purpose and conveyed to the packing-bench.

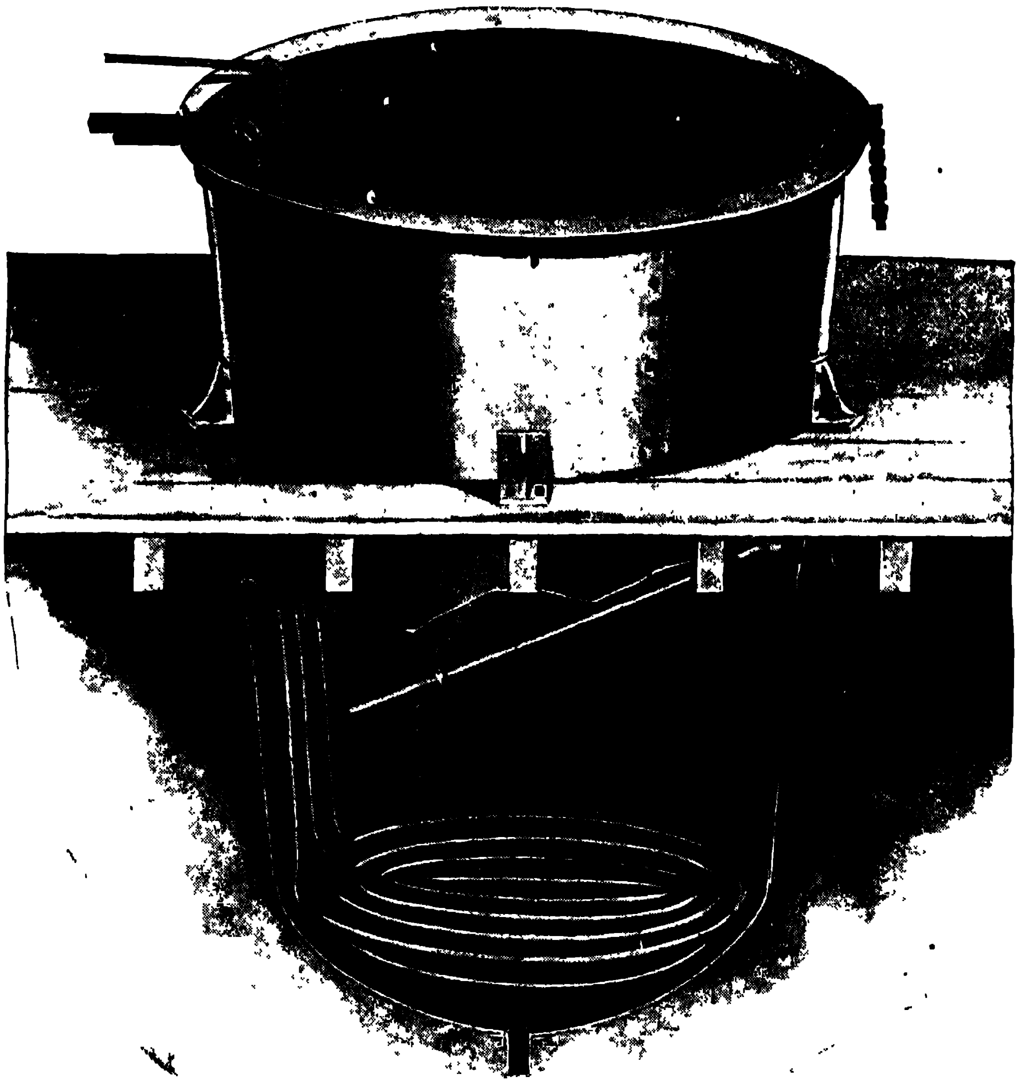


FIG. 42.—Dished Tapering Kettle with Open- and Closed-Steamcoils.

The Soap-kettle. — The contrast between the primitive fire-heated cast-iron boiler supported and enclosed by masonry and the present riveted, sheet steel and steam heated kettle, is typical of the advance in the art of soap-manufacture that has taken place during the past forty years. The modern soap-kettle is of circular, square, or rectangular cross-section and of varying capacity according to the extent of manufacturing operations. Cylindrical kettles may be of either dished or flat bottom. Kettles of square or rectangular cross-section may be either of flat bottom or with bottom inclined to one side. Cylindrical kettles vary in diameter from 5 to 25 feet and in depth from 8 to 35 feet with capacity ranging from 10 to 300 frames. Kettles of both shapes are made of two or more courses of steel plates, diminishing in thickness from $\frac{3}{8}$ inch on the bottom and $\frac{3}{16}$ inch on the lower course to $\frac{1}{8}$ inch on the upper course. Dished kettles discharge through the bottom and side; flat-bottomed kettles discharge as a rule through the side alone; for which purpose with dished cylindrical kettles a plate or ring with usually a 3-inch opening is riveted to the cone of the dish and with dished square kettles a 3-inch flange is riveted close to the lowest edge on either side or bottom, for the discharge of waste lye or the transfer of the contents of one kettle to another. For the discharge of good soap to the crutcher a 3-inch flange is riveted to the side of both cylindrical and square kettles for the attachment of a 3-inch cock on the outside and a swing-joint pipe of larger diameter on the inside. These two

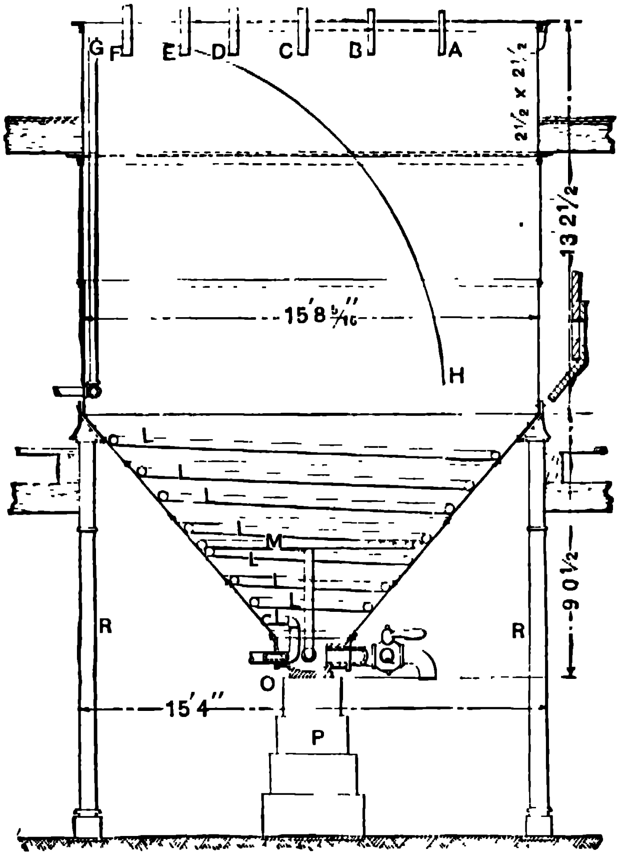


FIG. 43.—Soap-kettle, Cone Bottom.

openings comprise all that are necessary to a soap-kettle and both as a rule are 3 inches and sometimes 4 inches in diameter, according to the size of the kettle.

Unlike cylindrical kettles, square kettles tend to bulge from the weight of the contents and require to be securely stayed or braced. The contents of cylindrical kettles boil and cool more uniformly than do those of square kettles, unless steam piping for the latter extends into the corners which, as a rule, are rounded on a curve of 6 to 12 inches radius. Cylindrical kettles are to be desired in preference to square kettles in every case save that of economy in floor space. Unless well protected from rapid radiation of heat, kettles should be jacketed either with wood, an air-space separating wood and kettle, or with asbestos covering. Excessive heat in floors through which the kettles extend also requires that they be covered. The bracing and support of flat-bottomed kettles are cheaper and simpler than with dished kettles, which advantage extends also to their pipe attachments, but the former are not so readily cleaned, which, as a rule, is necessary three or four times a year, according to conditions and procedure. The circumference of cylindrical kettles at the top should be reinforced with a band of iron riveted on; with square kettles

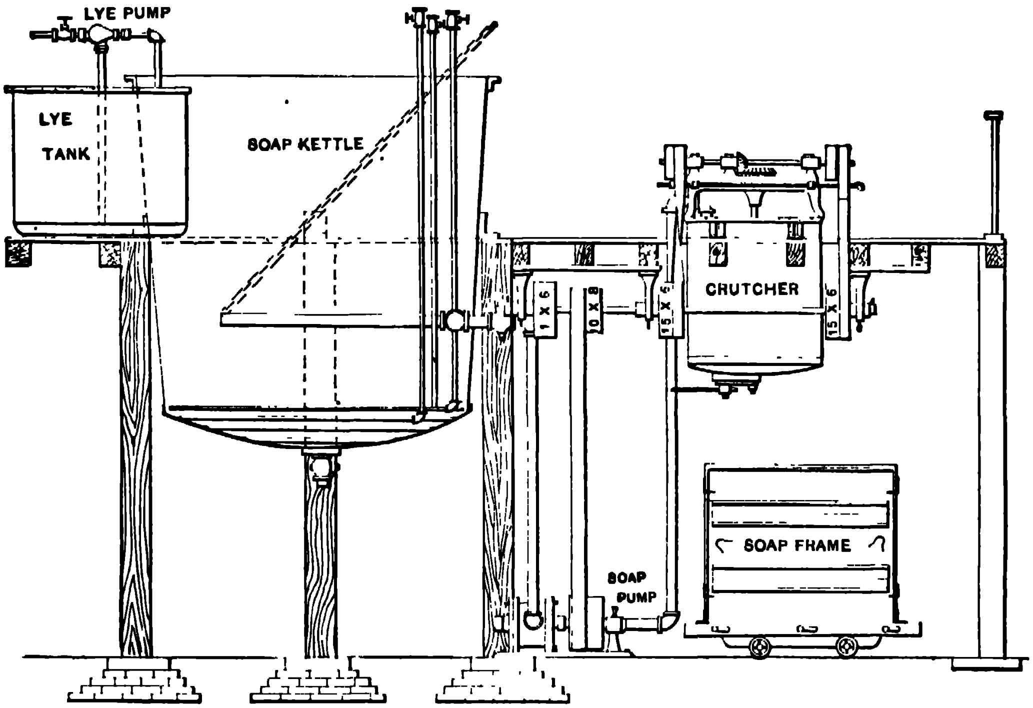


FIG. 44.—Kettle-plant.

angle iron should be used for the same purpose. With dished kettles no waste-lye discharge-pipe on the inside is necessary; with flat-bottomed kettles such a pipe is necessary and should extend to the middle, where the end should look down and almost touch the bottom; an elbow on the end may be used. The top of the kettle should extend at least four feet above the level of the kettle-room floor. Kettles should be located to admit of ample passageway between them and in no case so near together that the contents of one kettle may boil over into the other.

With the discharge of good soap by gravity to the crutcher, which is the most satisfactory arrangement wherever practicable, kettle connections are made solely through the lye pipe which is usually not less than 3 inches in diameter.

The lye discharge-pipe from each kettle connects with a main of the same diameter, a 3-inch cock being located between the kettle and the main, through which, by opening or closing the desired cocks, material in one kettle may be transferred to another by gravity, the respective levels permitting, or to a storage tank at lower level in case of waste lye. Where material is pumped from one kettle to another the pump withdraws it from the bottom of one kettle, elevates it and discharges it through a pipe of the same diameter into the desired kettle, or storage-tank. The piping system comprises a main to which each kettle is connected by means of a pipe carrying a cock which we have called the waste-lye discharge-pipe, in contradistinction to the soap discharge-pipe, although it may be used for the transfer of whatever material may be in the kettle, provided it be of suitable consistency to pump. The main is connected with a rotary pump which receives material from the main, elevates it and discharges it into another main suspended over the tops of the kettles in the kettle-room, which, in turn, by means of suitably placed cocks, attached by tees and short nipples, discharges it into any desired kettle. This arrangement constitutes the simplest, cheapest, and most convenient system of kettle connections. Between the main and rotary pump a cock is placed, which it is necessary to close in transferring material by gravity from one kettle to another or to a storage-tank. In pumping material from one kettle to another, it is necessary to open the cock connecting the kettle, from which it is desired to pump, with the main; also the cock connecting the main with the pump, and as well the cock in the main over the kettle into which it is desired to pump. After such a transfer of material it is necessary to blow out with steam any material remaining in the pipe, otherwise on cooling it will solidify and clog the system. Free ends of the main are closed with plugs which may be removed for cleaning the pipe. Between the cocks and the kettles and at a suitable place on the main, ¼-inch holes are bored for the insertion of steam-pipes, whereby after using the main, or if for any reason while pumping the flow of material is interrupted, steam may be blown both through the lye discharge-pipe

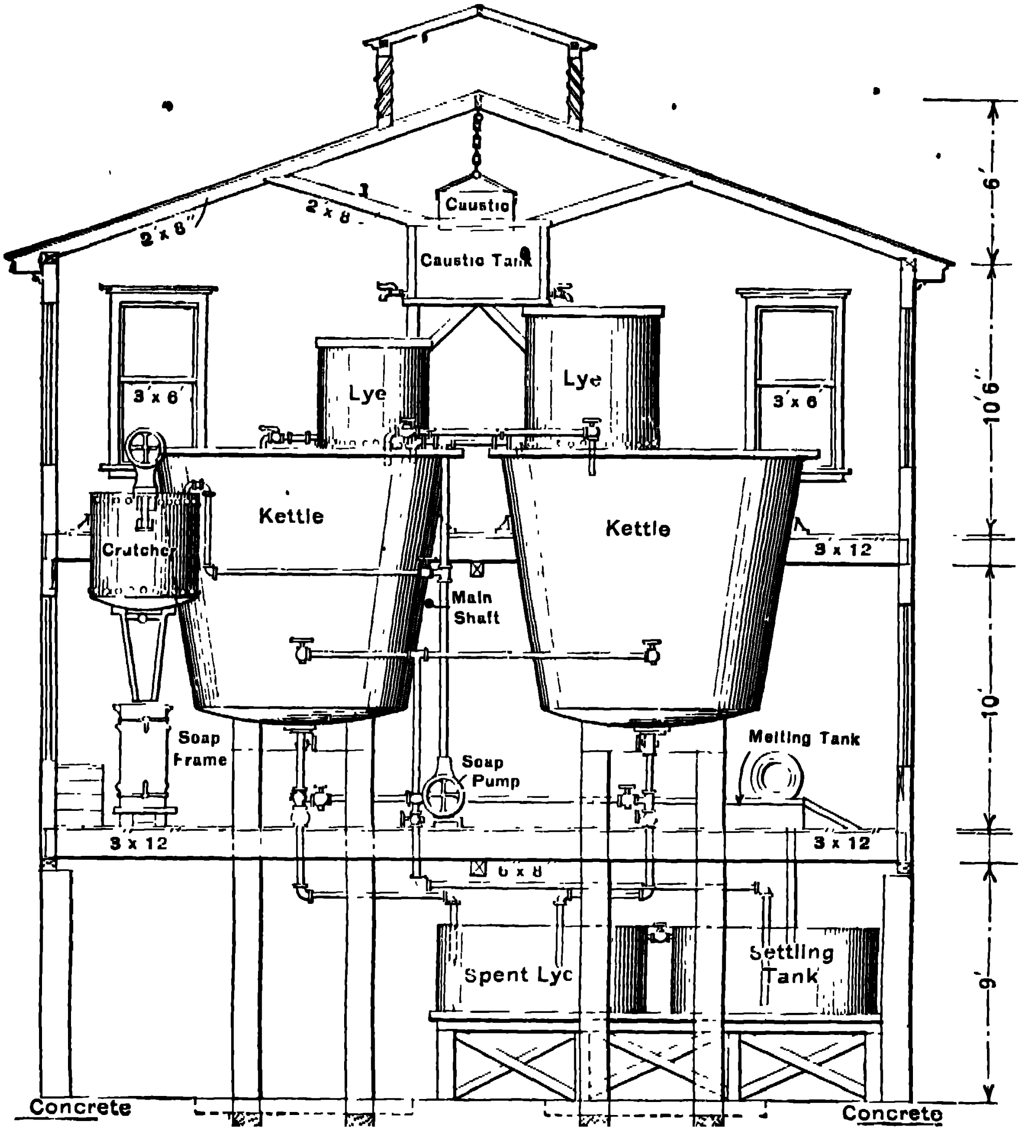
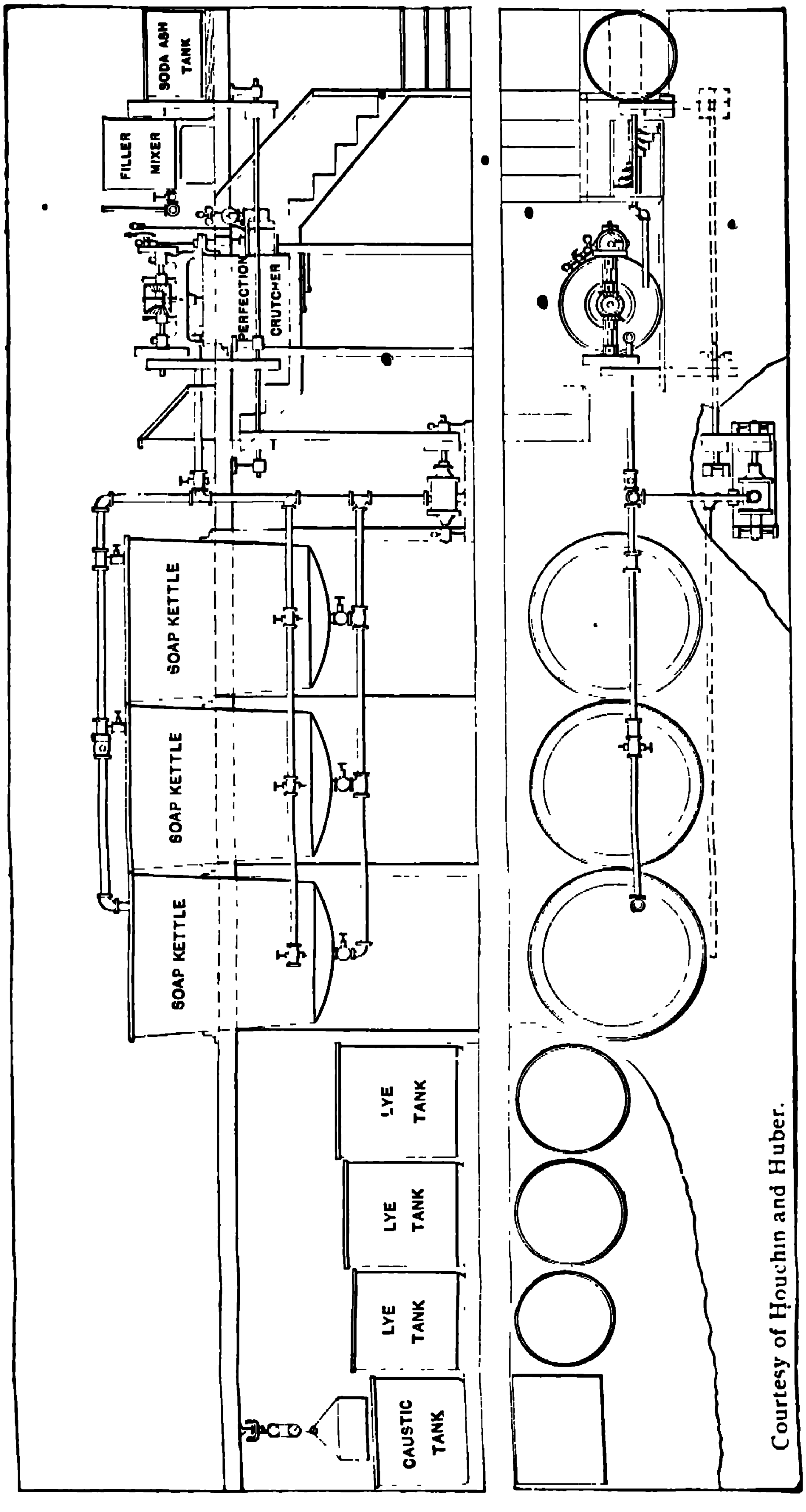


FIG. 45.—Kettle-plant.



Courtesy of Houchin and Huber.

Fig. 46.—Kettle-plant.

back into the kettle and through the main in the opposite direction.

Soap-kettles may be heated by means of either open- or closed-steam coils, or by both. Closed-steam coils for the boiling of soap may be used, but by many are not considered essential. The simplest, cheapest, and, as a rule, a satisfactory means of heating, is by the sole use of an open-steam pipe, which is shown in its rudiments in Fig. 47. This may take the form of a coil as shown in Figs. 42 and 51, but in its cheapest and equally satisfactory form for flat-bottomed kettles it is of the criss-cross type, the main pipe being almost equal to the diameter of the kettle and connected with arms extending at frequent intervals on both sides from the diameter to the circumference of the kettle. The construction of a dished kettle with use of both open and closed steam is shown in Fig. 43. It is supported by a heavy masonry pier *P*, and four cast-iron columns *RR*. The pipes *A*, *B*, *C*, *D*, *E*, and *F* deliver respectively water, weak and strong lyes, and first, second, and third grades of stock.

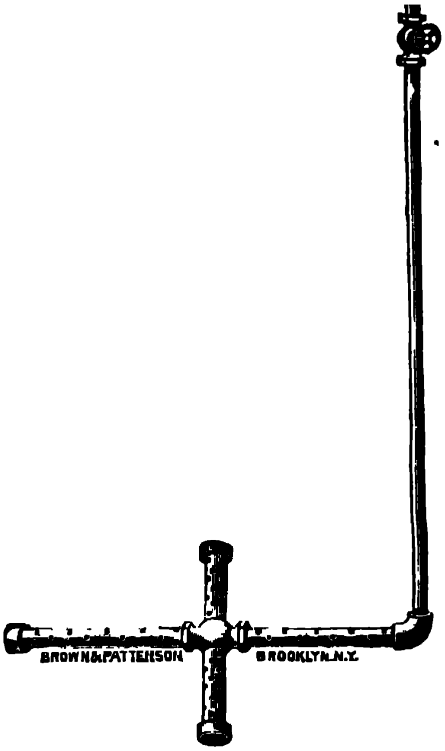


FIG. 47.—Criss-cross for Open Steam.

O is a cast-steel base, and *LL*, etc., is a 2-inch closed copper steam-coil; *M* is a 2-inch copper perforated steam-pipe; *G* is an iron swing-joint pipe, through which soap is pumped to the crutchers. It is attached at *I* by means of short nipples and two elbows, and its upper end can be moved through the arc *GH* so as always to draw from the surface. Waste lye is removed at the bottom represented conventionally by he cock *Q*. In Fig. 48 are shown the plan and section of a kettle-house, with elevator, conveyer, and distributing cone for the handling of rosin.

While the method of kettle construction and arrangement of kettles, as shown in Figs. 43 and 48, are by no means general, they represent conditions prevailing in a large plant. In Fig. 49 is a view of a kettle-room where square kettles are used.

Continuous lengths of coiled pipe for either open or closed steam are of high initial cost and expensive to repair if broken. They may be cheaply and efficiently substituted by built-up coils made of short lengths of pipe connected by 45° elbows. Such a coil may be lengthened as desired, and if broken the damaged section can be cheaply and quickly replaced by a new length. This arrangement is shown in Fig. 50. There is much diversity of opinion as to the use of open- and closed-steam coils. With the former, caustic lyes in general should be of slightly higher density to allow for dilution by condensed steam, otherwise there is no essential difference in the manipulation of the kettle by the two methods. Many soap-makers recommend the use of wooden covers to kettles to prevent too rapid cooling during settling. Where overhead ventilation is not practicable galvanized iron hoods may be suspended over the kettles for the removal of heat and vapor. Both soap- and waste-lye pipes are ordinarily 3 inches in diameter. It is better to have kettle connections too large than too small, and in every case ample facilities for cleaning should be provided. Asbestos-packed cocks are most economical and should have a flange-union

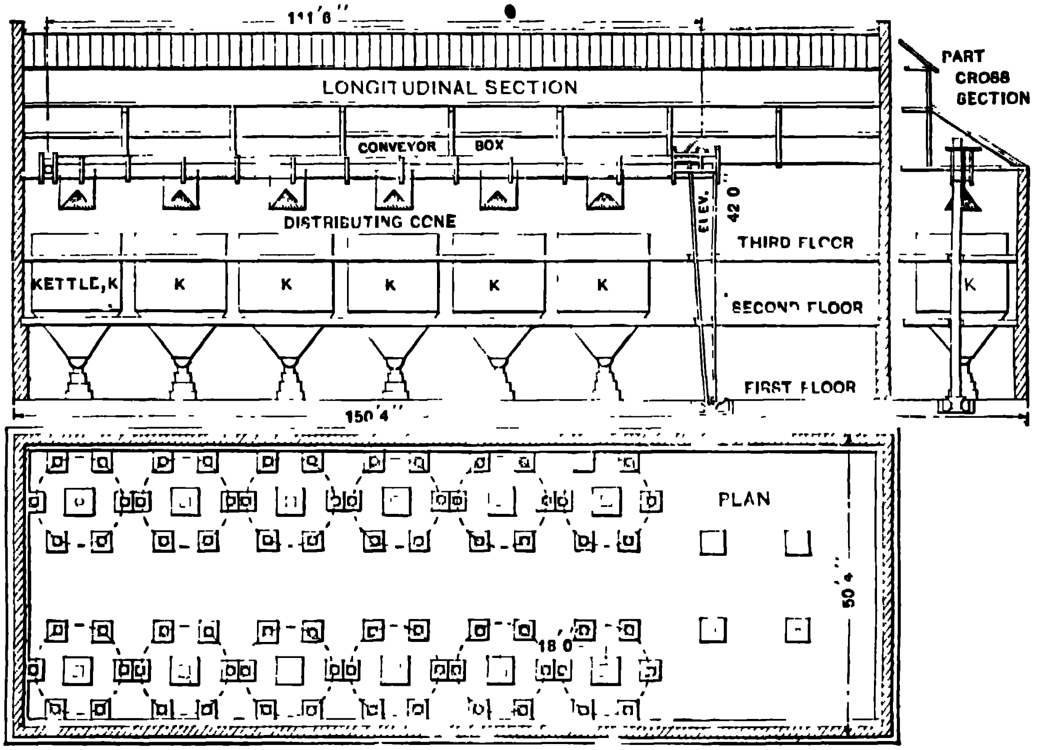


Fig. 48.—Kettle-plant.

on each side, so that it may be removed without difficulty should occasion require. Water-pipes may be of galvanized iron, but all others should be of standard black pipe. Steam blow-out pipes for cleaning the piping system are usually of ¾ inch diameter. The flange connection to the kettle, bearing on the inside the swing-joint pipe and on the outside a short nipple and 3-inch cock for the soap-

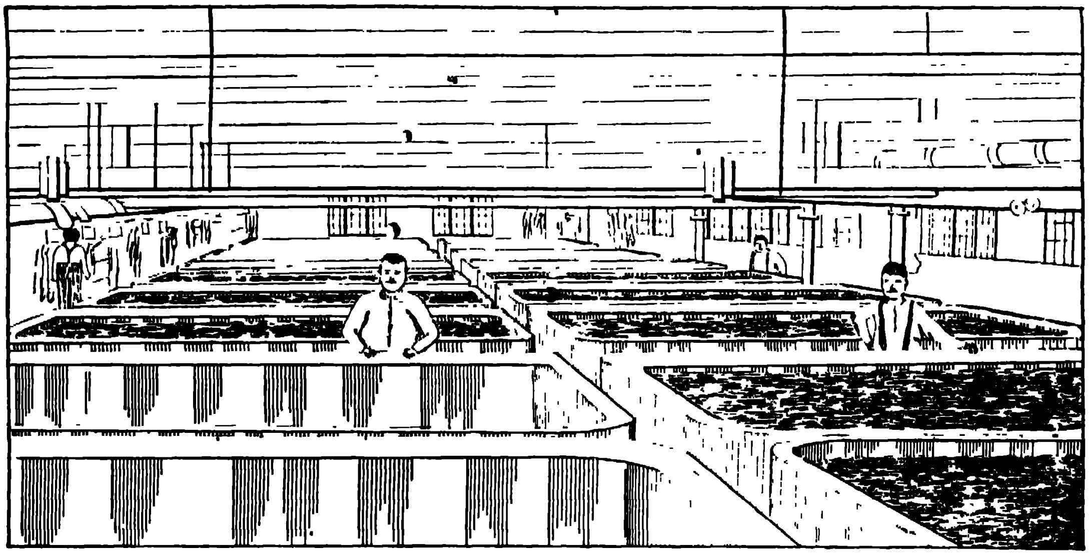


FIG. 49.—View of Kettle-room —Square Kettles.

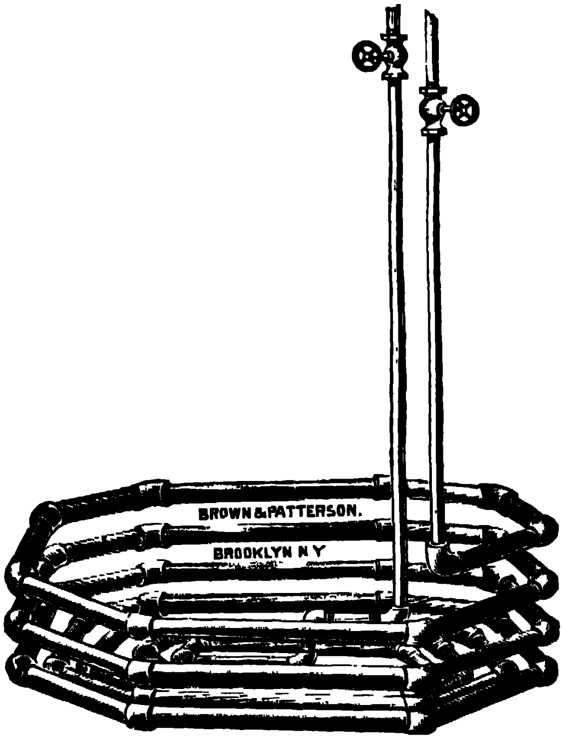


FIG. 50 —Built-up Closed-steam Coil.

discharge, should be at a sufficient distance from the bottom to permit of the discharge of the entire amount of good soap by gravity, and not require dipping as the nigre is approached. It should also be provided with a steam blow-out pipe. For a flat-bottomed kettle 16 feet deep, the soap discharge-pipe should be at least 3 feet from the bottom of the kettle. The kettle-room should be well ventilated and well lighted, and preferably located on the top floor.

A cylindrical kettle 14 feet in diameter, 26 feet deep and dished 2 feet will yield 175 to 200 frames of good soap. A flat-bottomed cylindrical kettle 12 feet diameter, 16.5 feet deep, will yield about 50 frames of good soap, and with ordinary working for a daily output of 500 cases at least seven will be required.

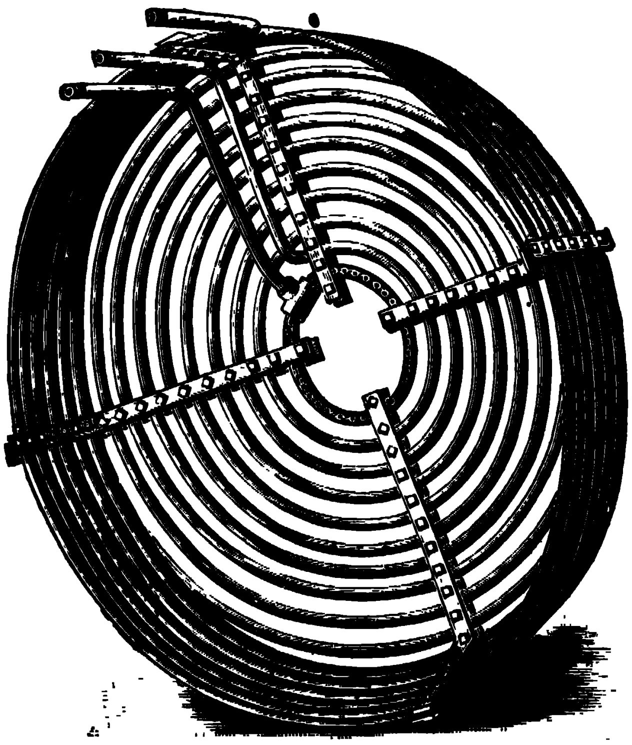


FIG. 51.—Closed- and Open-steam Coils.

The Rotary Pump.—A rotary pump is a device consisting of one or more projections acting as plungers, fixed to an axle, and revolving in a cylindrical case, for lifting or forcing fluids.

Rotary pumps in general have a certain field, outside of which they are not desirable, but within its limits they have most decided advantages. A rotary pump is hardly suitable as a fire-pump, as the pressure in the discharge-pipe can hardly be raised to and maintained at 60 pounds; but rotary pumps have the one great advantage that with relatively little steam or power they will pump a large amount of liquid against a limited pressure. They find a field of general usefulness in the soap industry for transferring fluid raw materials and products. Several types are shown in the accompanying figures. They consist essentially of a heavy cast iron base supporting both pump and pulleys. The Hersey pump shown in Fig. 54, can be operated in either direction. In Fig. 55 is also shown the construction of the operating blade and cone, which form the only working part of the pump. When run in the direction indicated by the arrow, suction takes place at the orifice *A* and discharge at the opposite side, and vice versa when the pump is run in the other direction.

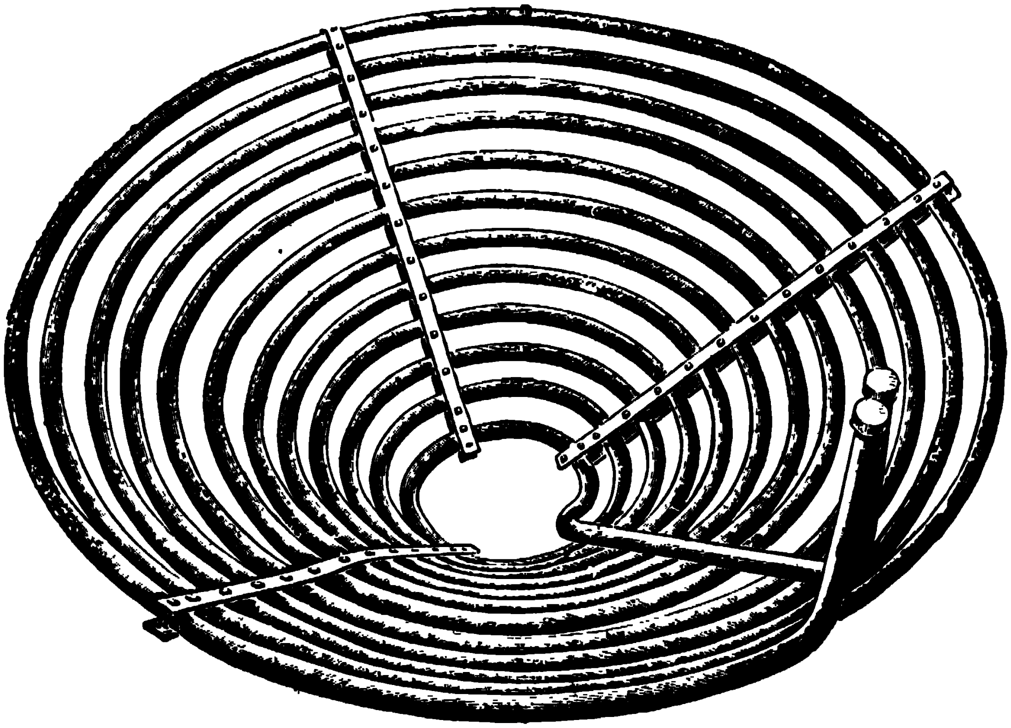


FIG. 52.—Closed-steam Coil.

Referring to the internal view shown in Fig. 55, a very good idea of the manner of its operation may be had. When turned in the direction of the arrow the blade *B* sweeps around, drawing the material in at *A* and forcing it out at *C*, the pump being emptied twice during each revolution. The blade *B* swings on a pivot, and the cone-shaped casting *D* maintains a division between the two

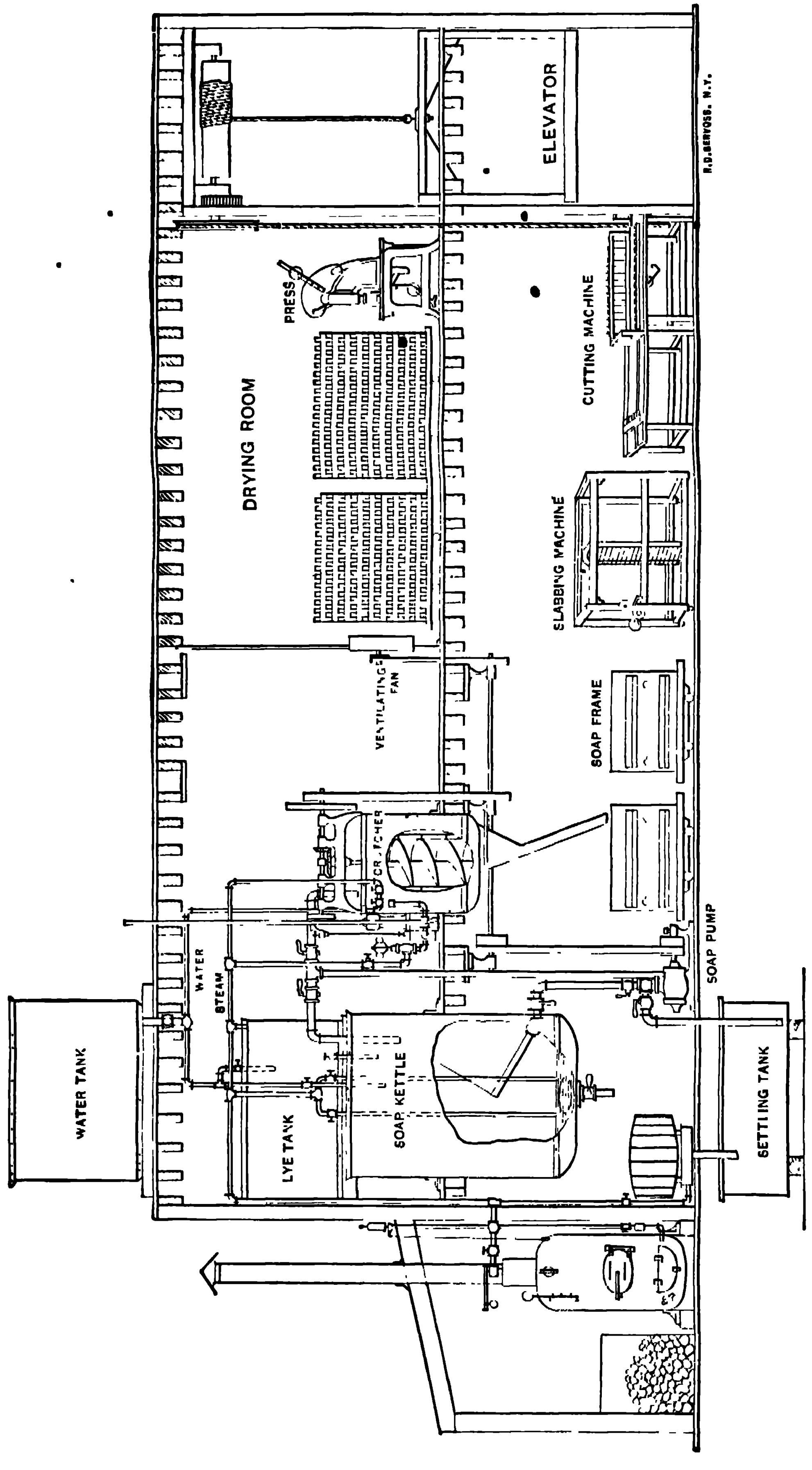


FIG. 53.—Soap-plant.

sides by contact with the cover, which is bolted to the main casting.

In Fig. 56 are shown the working parts of the Taber rotary pump, in which *A* is the shell or case of the pump; *BB* are the heads covering the ends of the shell; *C* is the piston that carries the valves; *DD* are the valves that pass through the piston in the ways *EE*, and as the piston is revolved pass in and out, back and forth through the piston, following the inside lines of the shell; and *EE* ways in the piston for the valve.

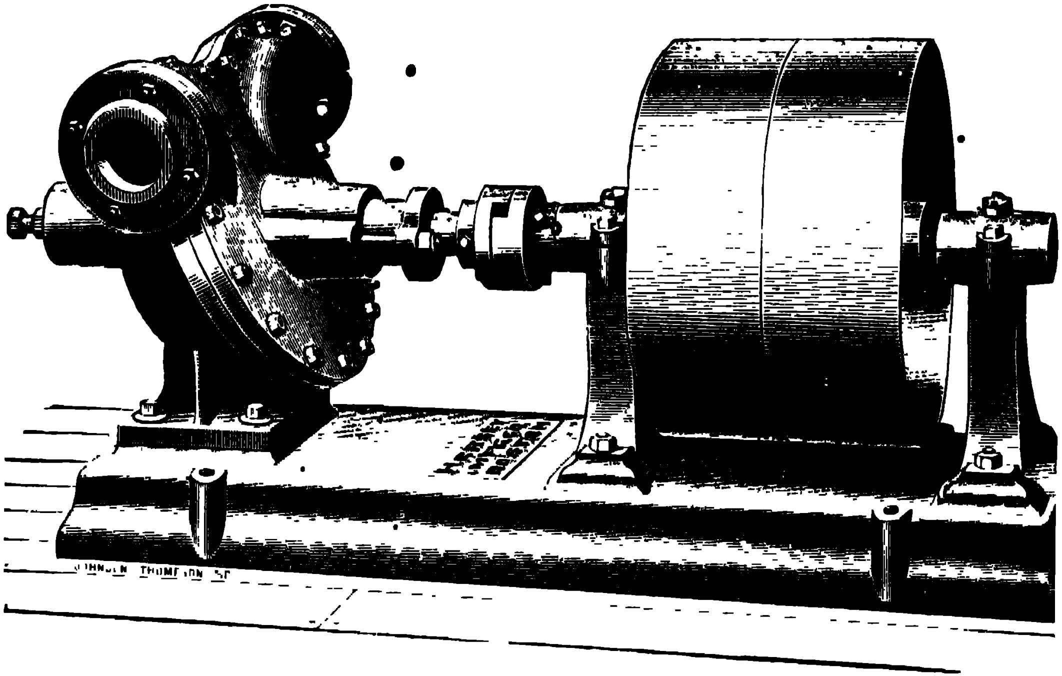


FIG. 54.—Hersey Rotary Pump

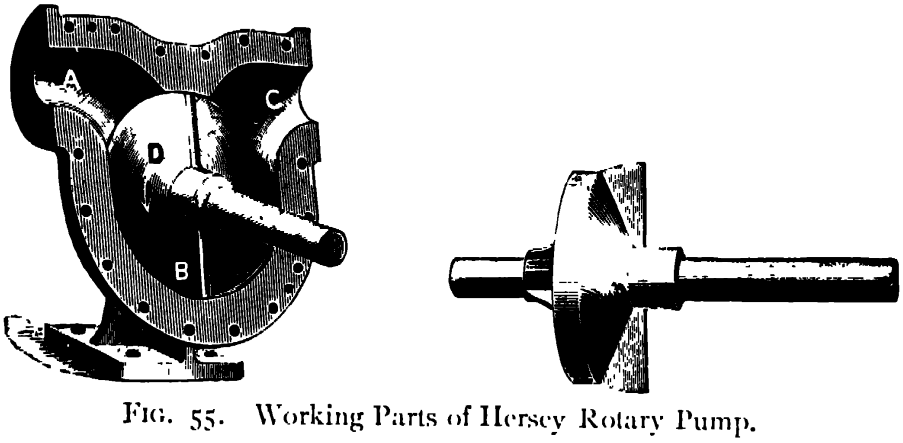


FIG. 55. Working Parts of Hersey Rotary Pump.

With the piston set tight against the shell at the point *F* between the points of suction and discharge, revolving the piston creates a vacuum on the inlet side and the pump is set in operation. The wear is chiefly on the ends of the valves *DD* and, when necessary, they may be readily replaced. The Taber rotary pump is also shown in Figs. 57 and 62.

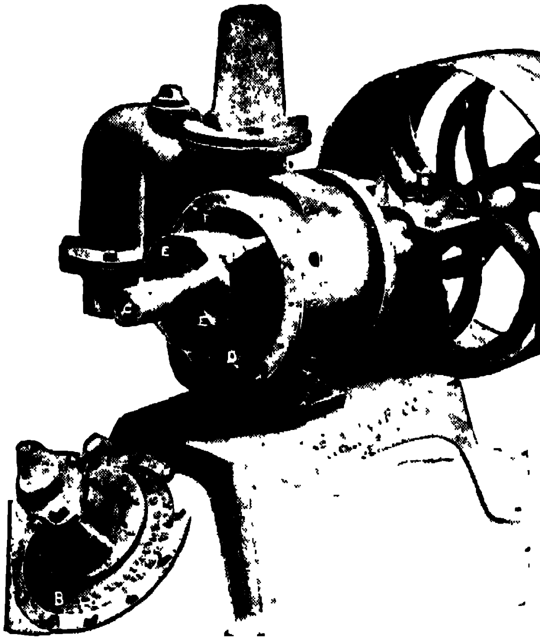


Fig. 56.—Taber Rotary Pump, showing Working Parts.

In the Lobee rotary pump shown in Figs. 58, 59, and 61, the four valves, Fig. 59, the vital part of the pump, are formed in two pairs, one pair having two parallel arms provided with longitudinal ribs or tongues, and the other pair having two parallel arms provided with longitudinal depressions or grooves, forming slide-ways

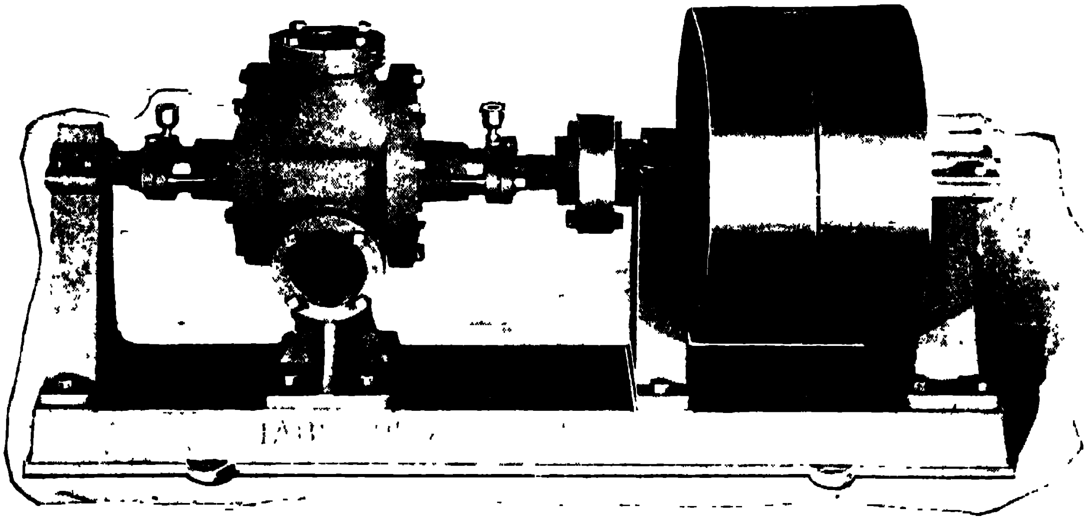


Fig. 57.—Taber Rotary Pump

in which the ribs or tongues upon the arms of the other valve seat and travel.

By this arrangement they are always kept in position to work, are self-adjusting, requiring no springs or cams to operate them whatever, they being positive in their action, and are like two solid valves, which prevents lateral motion, back lashing, and from getting wedged in the slide-ways of the piston.

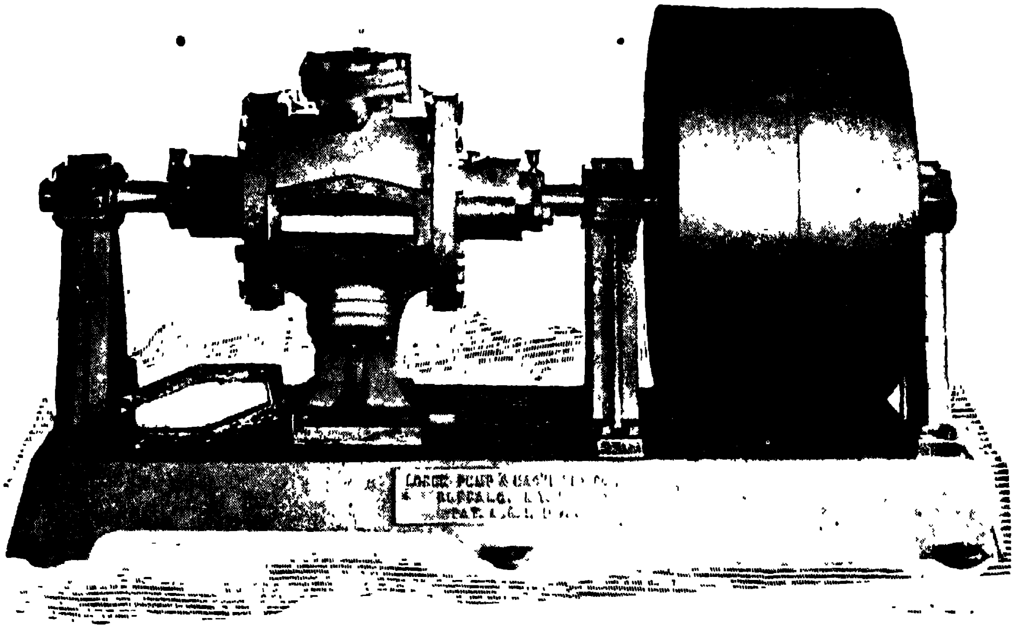


FIG. 58.—Lobee Rotary Pump

These two pairs or four valves are interchangeable, and the wear can be easily taken up by means of set-screws in the arms, which are the same thickness as the valves.

The piston which carries the four valves and through which they slide back and forth during their operation is made wide at both ends, which prevents it from breaking. The shaft of the piston is made of steel.

This extra width of piston is taken up by two half-moon pieces, which conform to the inside of the shell, and are bolted on to each one of the two heads of the shell. This prevents the valves from wearing against the heads of the shell. The wear on the half-moon pieces can be easily taken up by removing them from the heads of the shell and placing a strip of paper or other suitable packing between them and the heads of the shell.

The shell has a removable plate or hand-hole, Fig. 58, whereby ready access to all the working parts of the pump is possible without dismounting the pump or withdrawing the piston from the shell. All rotary pumps should not be located over ten feet above the surface of the liquid to be pumped, and should be placed preferably below the level in order that gravity may assist in the flow of the material to the pump, also that a steam-pipe be connected to the discharge side of the pump so that steam can be applied to blow out and clean the interior.

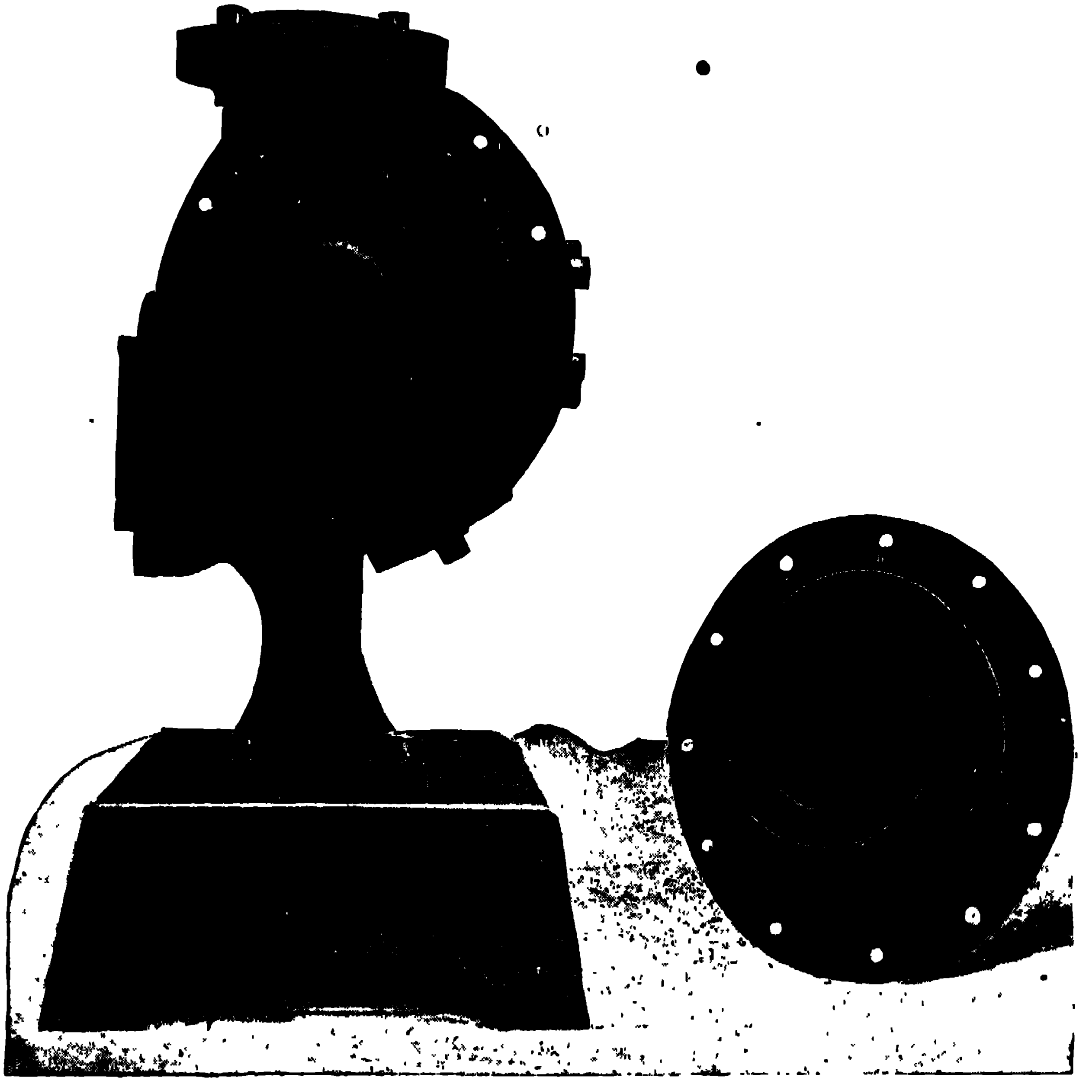


FIG. 59.—Working Parts of Lobee Rotary Pump.

Liquids may also be transferred by means of the ejector, Fig. 60.

This device is useful for transferring stock or waste lye from one tank to another above levels where gravity does not avail.

All sizes will lift liquids 25 feet and elevate them about 15 feet above the ejector with a steam pressure of 60 pounds.

If it is desired to elevate liquids a greater distance than 40 feet, the ejector should be placed near the liquid so that it can be forced by the ejector. In this manner liquids can be lifted about 50 feet with 75 pounds pressure, and about 70 feet with 100 pounds pressure.

The Strainer.—The ease with which insoluble foreign matter finds lodgment in the kettles and pipe system requires that some method to prevent its introduction and to secure its removal should be adopted. Stock as steamed from barrels should be strained; when pumped from the storage-tanks a swing-joint pipe should be used, or the outlet should be several inches above the bottom of the tank. Soap and waste lye should be strained before passing through the pump.

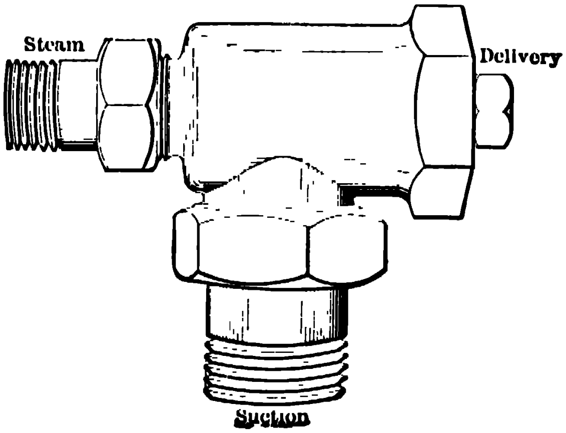


Fig. 60.—Ejector.

Soap-makers experience much trouble by foreign substances, such as nails, bungs, etc., being drawn into the pumps through the suction inlet, thereby clogging or injuring the pumps and causing annoyance and delay in the crutching and framing rooms.

Devices for intercepting material of this sort are shown in Figs. 61 and 62. With the strainer shown in Fig. 61, soap can be strained to any fineness desired by merely adjusting the rings. Where soap is discharged from the kettle to crutchers by gravity through a trough, netting suffices; but where pumped the soap should pass, where handled in large amount, through a specially built sieve. One form consists essentially of a cylindrical perforated iron vessel suspended within a cast- or sheet-iron tank, about the size of a half-barrel, with tight but detachable cover. The soap-pipe connects with the enclosed sieve and the soap is withdrawn from the bottom of the outer vessel after having passed through the sieve. Steam blow out connections are attached.

Filler-mixer.—Where liquid, semi liquid or solid substances are used in combination or separately in crutching soap, a device for their perfect mixing previous to their addition to the crutcher to be incorporated with the soap is shown in Fig. 63. Where crutching is an uninterrupted part of the factory procedure, devices for the expeditious, and so far as possible, continuous handling of filling material are to be desired. They should be labor-saving and the flow of materials should be aided by gravity. To expedite the crutching process and to secure thorough admixture of filling material, especially when of different specific gravity and consistency, a filler-mixer conveniently situated to secure the ends mentioned is recommended.

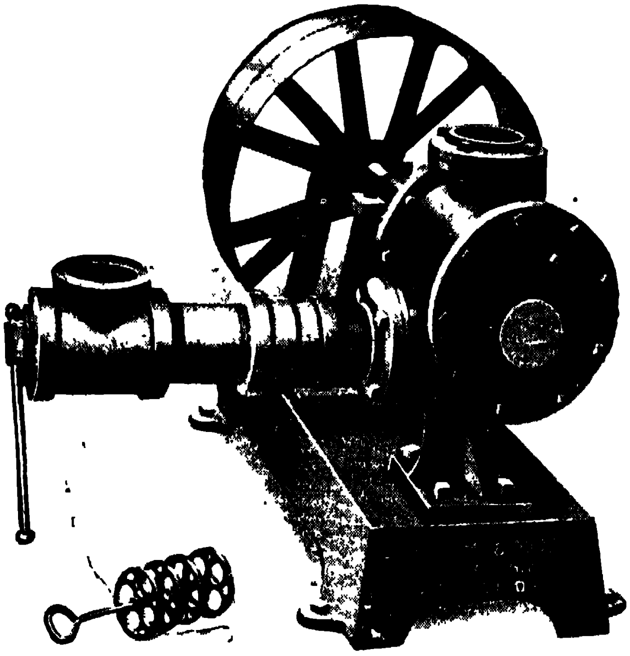


FIG. 61.—Soap-pump with Strainer Attached.

The Crutcher.—Mechanical soap-crutchers are an evolution of the simple hand crutch shown in Fig. 64, by laboriously working which up and down in the mass of soap contained in a frame, cooling of the soap was hastened, whereby either separation of the nigre was prevented, or filling material incorporated. The modern power-driven crutcher consists essentially of a cylindrical tank, as a rule of about 1200-1500 pounds capacity, made of sheet-steel or boiler-iron, either plain or jacketed, and supporting the driving gear

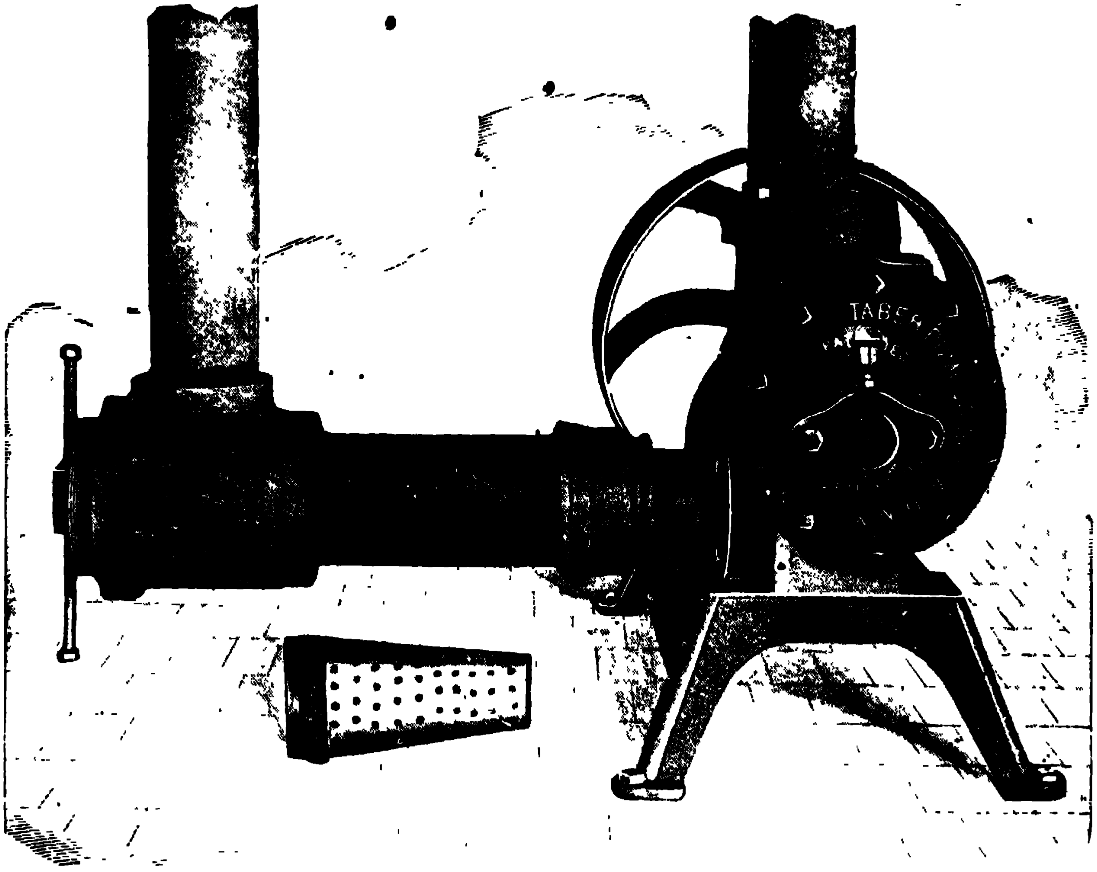


FIG. 62.—Soap-pump with Strainer Attached.

of the agitator, which may be either vertical or horizontal, and which consists of either blades or arms attached spirally to the horizontal or vertical shaft, or a screw enclosed within an inner, vertical, concentric cylinder, whereby material added to the soap and the soap are reduced to a homogeneous consistency. In the crutcher shown in Fig. 65, instead of carrying its contents this machine simply crutches, keeping the soap in a body. The wings are constructed in a spiral form and work as a double-acting screw, raising and mixing the heaviest material from bottom to top.

In the type of crutcher shown in Figs. 67, 68, 69, and 70, mixing is effected by the movement of the mass of soap by means of a screw attached to the vertical shaft and moving in an inner concentric cylinder. In crutching, the movement of the soap is upward in the cylinder and down between the cylinder and the sides of the crutcher. In discharging the contents the motion is reversed.

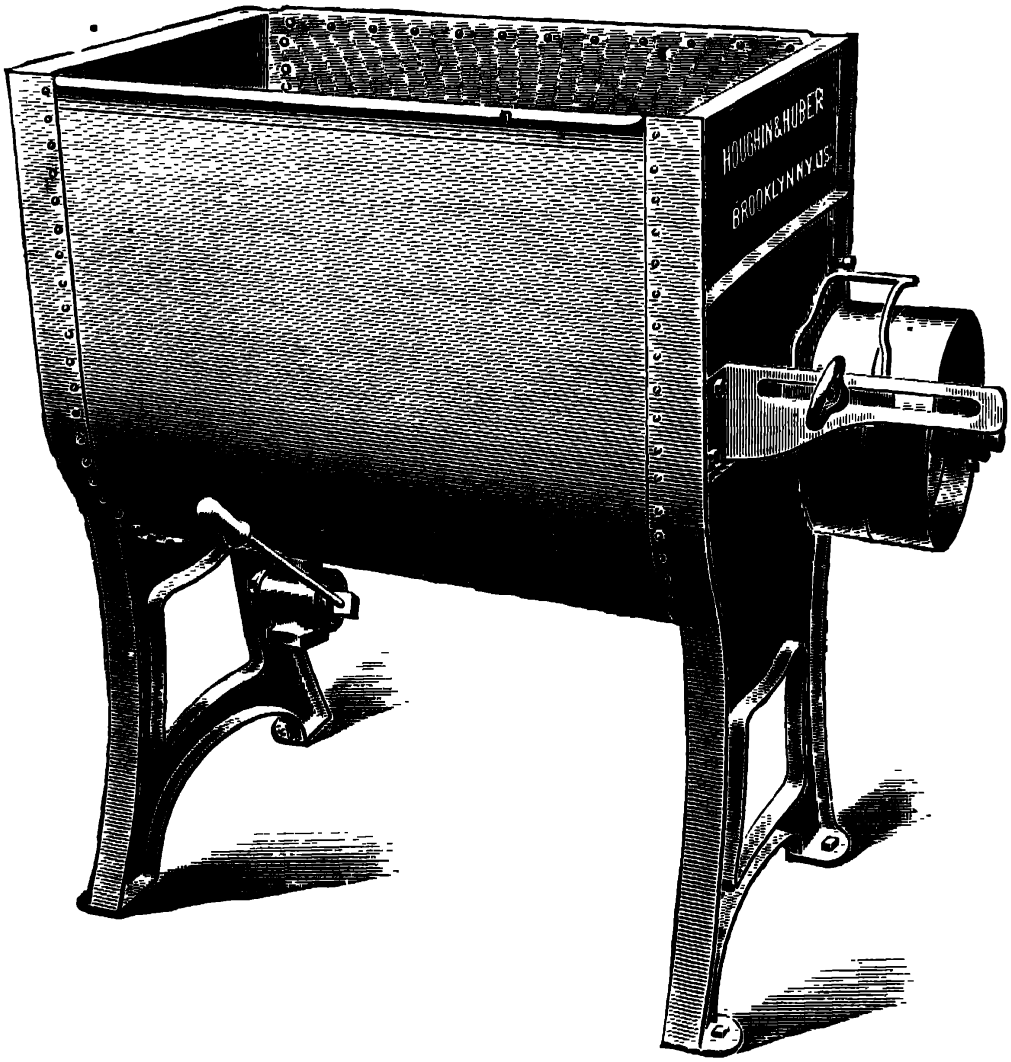


FIG. 63 - Filler-mixer.

By jacketing the inner cylinder, Fig. 69, or the sides of the crutcher, or both, and introducing steam, the machine can serve as a remelter.

In the type of crutcher shown in Fig. 66, the shaft carrying the blades is horizontal and the soap is discharged at the end at the bottom. By the spiral insertion of the blades the movement of the soap is directed toward the end of the crutcher. As with all crutchers, two styles are commonly made: one of which has a steam jacket, the other without a jacket. In the latter the soap is taken directly from the kettle and thoroughly mixed or crutched in the state in which it is received. In the jacketed machine the soap can be kept at any temperature desired during the operation of crutching. Hard and dry soap can be crutched without difficulty by letting steam into the jacket and warming it up, and by substituting cold water for steam the soap can be cooled.

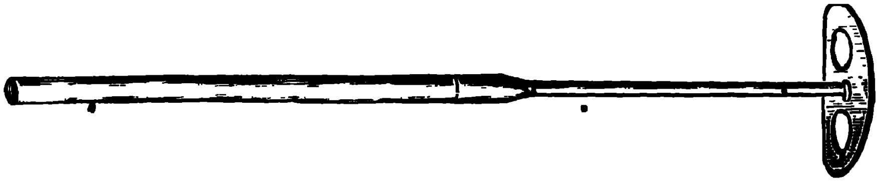


FIG. 64.– Hand Soap-crutch.

The jacketed machine, Fig. 66, is constructed with divisions or vanes at intervals, so that a perfect circulation is provided, leaving no places for warm water to remain without circulation. It is provided with steam and water inlet-pipes, escape-pipe for steam, and a valve for letting water out of the jacket. A spout is arranged at the bottom of the machine so that the soap can be run directly into frames placed underneath.

The paddle-shaft should run about 45 to 50 revolutions per minute, and should turn in the direction to work the soap toward the outlet end.

The soap should cover the paddles about two inches before starting the machine.

The paddles should not be running when the crutcher is being emptied into frames unless the soap is running very slowly, otherwise the soap will be forced out too rapidly.

The machine is cleaned by adding four or five pails of strong brine and running it four or five minutes.

In the jacketed machine the escape steam-pipe should have no valve, but should be left free.

Water should always be let out of machine when not in use to prevent freezing and bursting the jacket.

Steam should not be let into the jacket until it is free of water, or it is liable to strain the jacket.

With the crutchers shown, Figs. 67 and 68, driven respectively by belt and by direct-connected engine, mixing is effected by means of a vertical screw enclosed in a jacketed cylinder, sides and bottom of the crutcher also being jacketed or plain, as desired. With the belt-driven crutcher reverse motion is obtained by use of an extra pair of pulleys carrying a crossed belt; with the crutcher driven by belt-connected engine reverse motion is effected by means of a clutch. Reversing is necessary at times with thick, heavy soap, and also to prevent splashing with a rapid movement of the conveyer when cold-process soap is made.

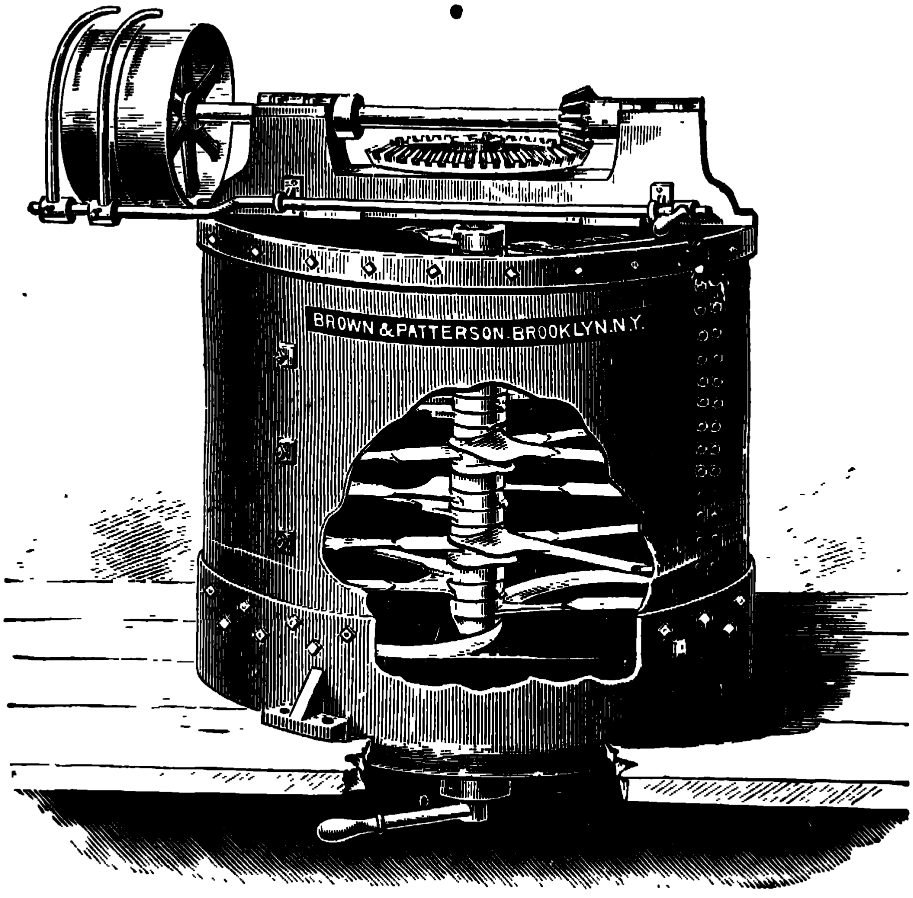

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FIG. 65.- Soap-crutcher.

In the remelting crutcher, shown in Fig. 69, the steam-jacket and inner shell are cast in one piece and has a large outlet in the centre of the bottom for the discharge of the contents. A steam-heated radiator, composed of a series of cylindrically arranged pipes having open spaces between them, is placed in the centre. Through this radiator steam passes directly to the jacketed part of the kettle, which can be cut off from the steam supply so that the inner cylinder only has steam. A conveyer-screw is placed in the centre of this radiator, which surrounds the screw. As soon as a portion of the soap is melted, the screw is set in motion, thereby lifting the soap up and dumping it over the top of the casing surrounding the screw, the centrifugal force forces it out of, or through the open spaces left between the pipes. The large scraps are carried up and are wedged in between the open ports at the upper end of the radiator. The constant motion of the screw shears the pieces off, and thus, in comparatively short time, the largest scraps are completely cut up, and the soap melted and crutched ready for framing. Owing to the open spaces left between the pipes composing the radiator, there is no splashing of the soap, however fast the conveyer-screw is worked. Moist steam may be passed at will through the soap scraps, etc., to moisten them, if necessary. If desired, cold water may be passed into the jacket and radiator to facili-

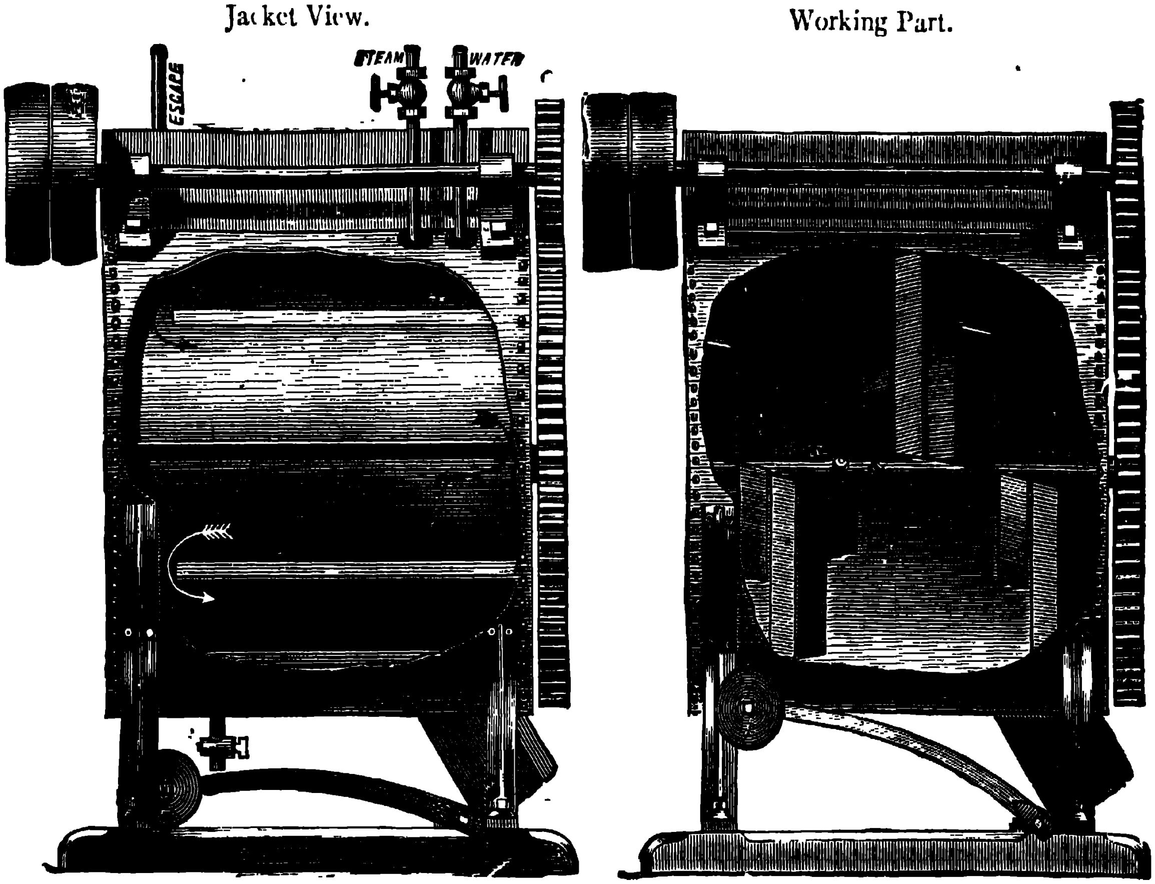

Jacket View. Working Part.

FIG. 66.—Strunz Crutcher.

tate the cooling of the soap. The conveyer-screw may be worked with a belt or driven by direct connected engine. The conveyer-screw may be worked forward or backward by merely shifting the crutch that drives the bevel gearing.

In Fig. 70 are shown two crutchers of the inner cylinder-screw type, connected with a 14-horse-power engine, all supported on one iron frame. The two crutchers are driven in the centre with one large pulley, 24 inches diameter by 6 inches face. Each crutcher has a clutch on the back of the pinion-hub, so that either machine

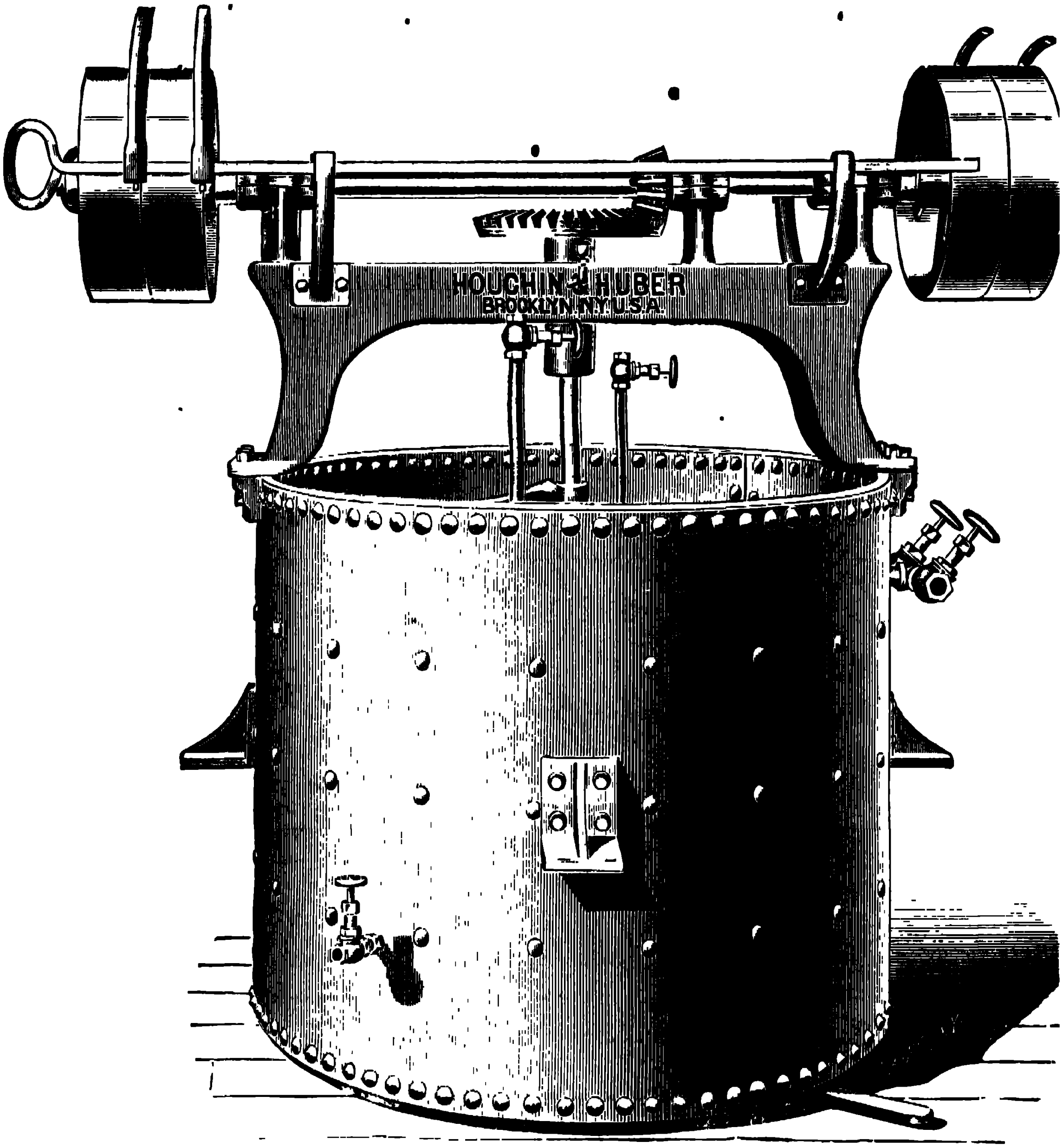


Fig. 67.—Soap-crutcher, Belt-driven.

can be thrown in or out of gear as desired. The horizontal shaft runs clear across the two mixers and takes in all the bearings on the two yokes. Attached to the top of the inner

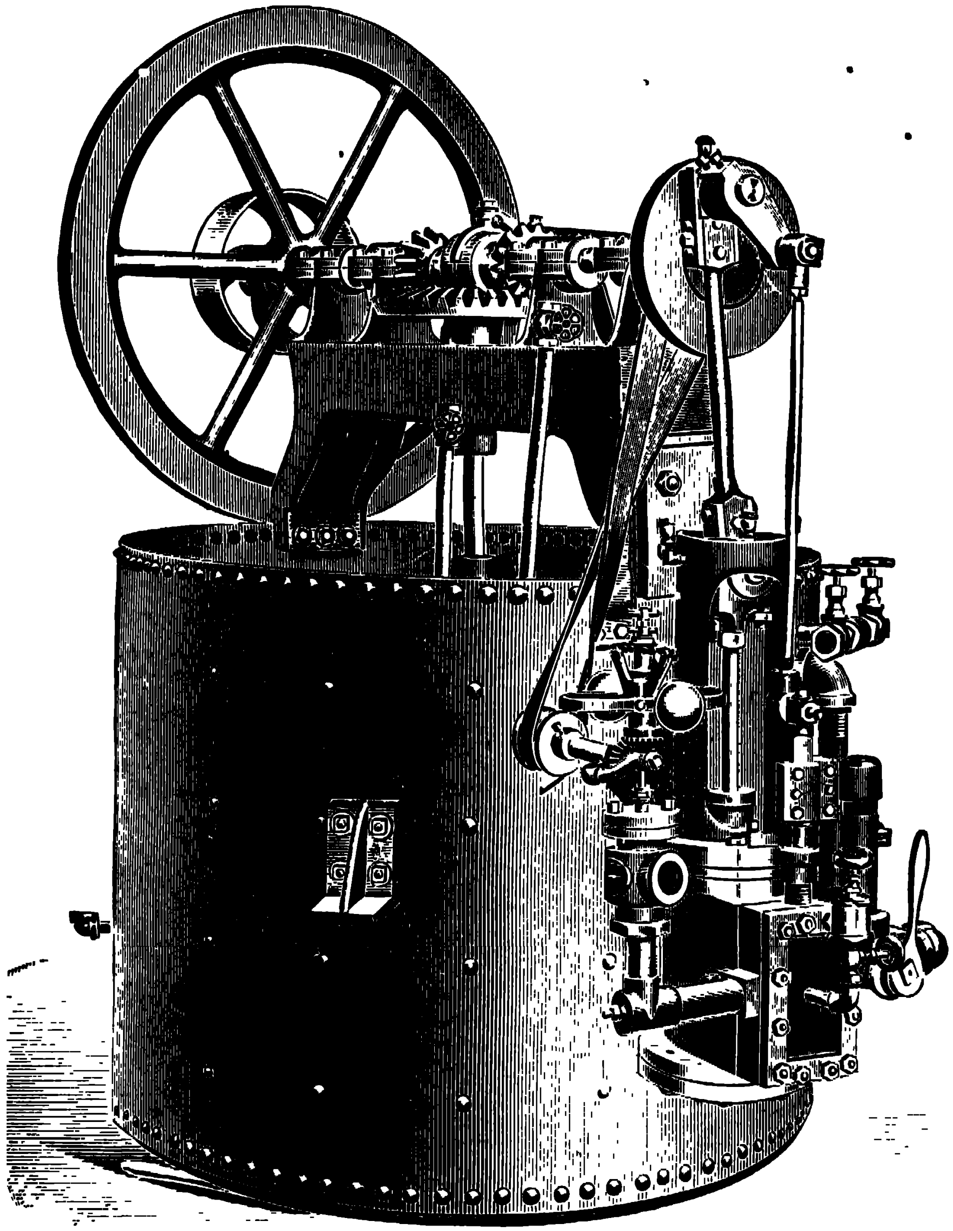
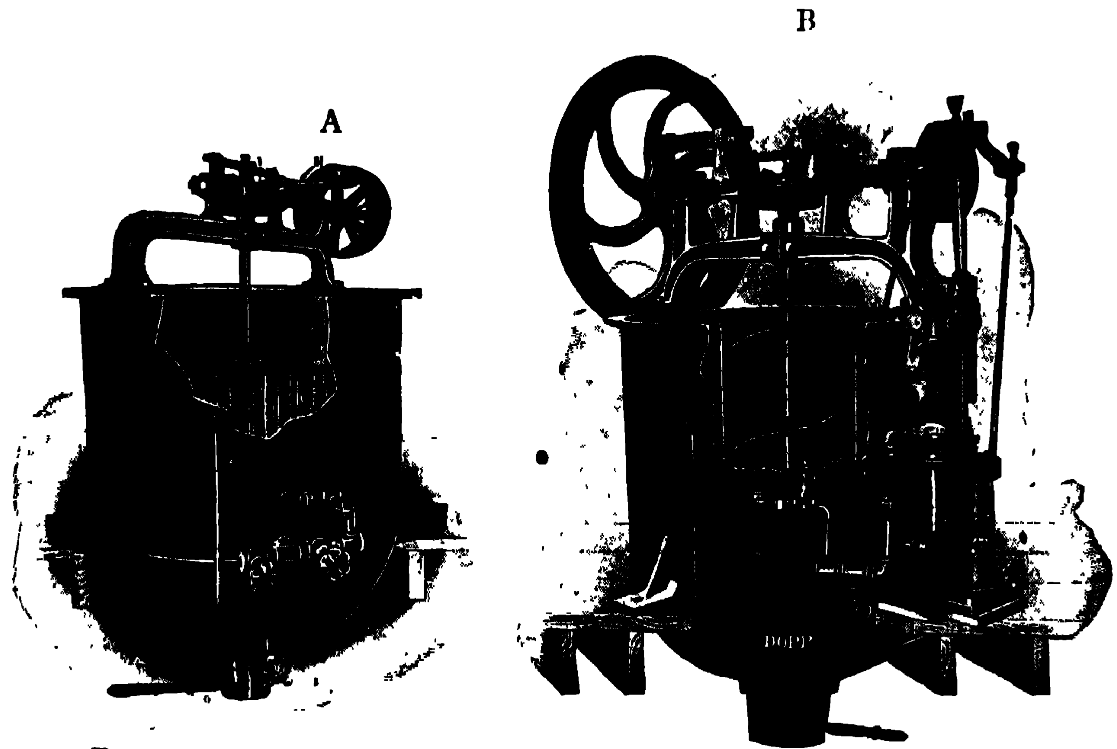


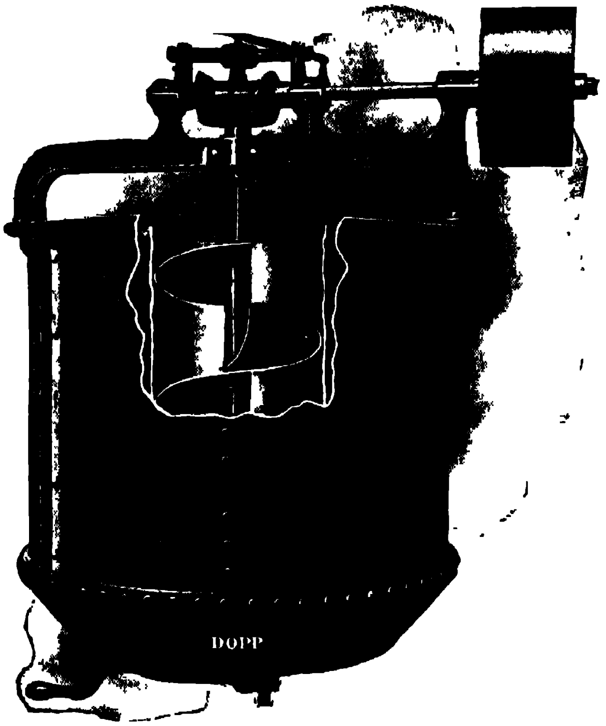
FIG. 68.—Soap-crutcher, Driven by Direct-connected Engine.

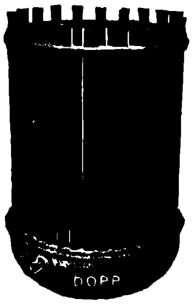
cylinder is a strainer to be used when scrap is remelted. The strainer is designed to retain the scrap within the jacketed inner cylinder and to allow the liquid soap to pass through, thus causing the scrap to melt more readily. The strainer



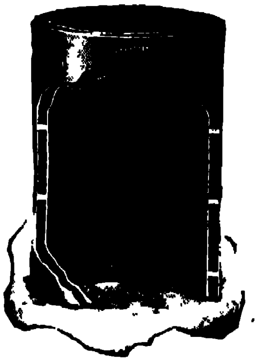
Remelting Belt-drive.

Remelting Engine-driven.





Pipe Radiator.



Plain Crutcher or Mixer.

Shell Radiator.

FIG. 69.—Crutchers.

is made in two parts and may be attached and removed with ease. In discharging the contents of the crutcher, crutching is stopped and the mixed soap is allowed to fall by gravity until the machine is partly emptied; then reverse motion, is applied whereby the movement of the screw directs the remaining soap downward and out.

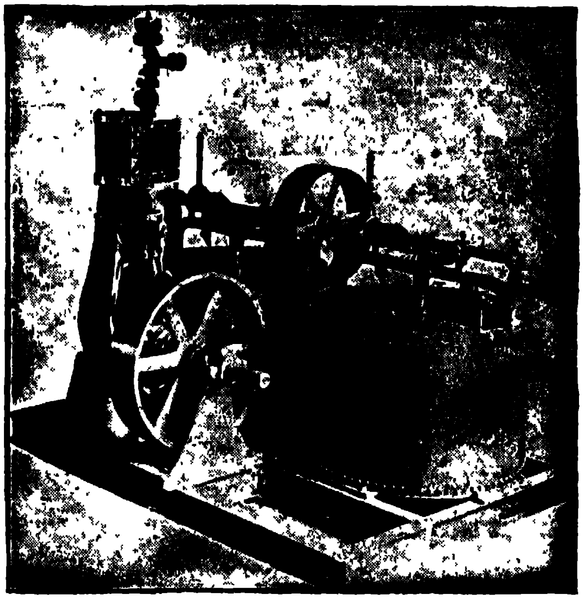


FIG. 70.--Crutchers.

Use of Crutcher.—Crutchers are used in soap-manufacture for incorporating alkaline salts with soap, for mixing soap and soda-ash for soap-powder, for mixing silex with soap for scouring-soap, for making all classes of cold-process soap, and to a limited extent in a small way, for semi-boiled soap, and for incorporating air with soap for floating soap. For each variety of use, not only is different manipulation required, but for a particular purpose one type of crutcher is better adapted than another. To avoid incorporation of air in soap the crutcher should be filled above the level of the agitator; to effect incorporation of air the motion of the crutcher should be reversed and the crutcher should not be filled so full, otherwise room for expansion on the incorporation of air will not adequately be provided. With soap-powder and sand-soap the same conditions with respect to air prevail; moreover the agitator should be powerful, as the material is heavy and tends to thicken. Cooling or heating to any desired temperature is effected as already described. Water of condensation should in steam-jacketed crutchers be promptly removed, otherwise seams will be weakened and leaks started.

The Frame.—A soap-frame consists essentially of a platform, commonly called a frame bottom, Fig. 71, upon which rests the mass of soap, and four upright movable sides, which sides, besides being fastened tightly together, must also be held or fastened tightly to the platform, each one of the said sides being situated rectangularly to its contiguous fellow-side and the whole apparatus being on wheels for convenience in moving.

The earliest frame was a box, tub, or vessel of any convenient shape. Later, wooden frames came into use closely approximating the dimensions of the present frame, and built up in sections tightly clamped from top to bottom; later cast-iron sides were used. The modern frame is of wood ends commonly, with sheet-steel sides reinforced by angle-iron or V-shaped strong-backs, with an average capacity of 1200–1300 pounds of filled soap. The inside dimensions of a frame of this capacity are: depth, 40 inches; length, 54 inches; width, 14.5 inches. The essential requirements of a soap-frame are, strength, lightness, freedom from leakage, ease and rapidity of stripping and setting-up and durability. The various styles made and used differ in construction and appliances designed to fulfill these requirements. They are made in sizes to suit the needs of each manufacturer. The most important dimension in a soap-frame is the width, for upon this depends largely the percentage of scrap at the cutting-table. Frame dimensions should be such that on slabbing and cutting, the maximum number of cakes of desired size may be obtained with a minimum percentage of scrap to be returned to the remelter or soap-kettle. The width of frames may be varied to adapt this dimension to the requirements of the dimensions of the cut-bar by inserting two or more grooves in one side of the frame bottom. With the frame side inserted in one groove, a slab cut lengthwise will yield a cake of certain dimensions with a minimum amount of scrap; with the frame side inserted in another groove, another cake of different dimensions with a minimum amount of scrap is produced. Frame ends and bottoms are of wood and as a rule about twice as many bottoms as pairs of sides are re-

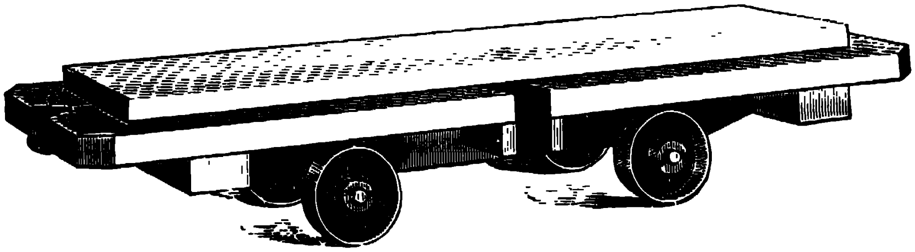


FIG. 71.—Soap-frame Bottom.

quired. For a daily output of 200 cases 60 complete frames are required, although this relation is subject to variation. After stripping, the soap remains on the bottom until cut; with an extra bottom, the sides and ends may be in use again before the original bottom is available.

Referring to the soap-frames shown in Fig. 74, *A* represents a frame made up of iron sides and ends. The sides have four angles across, with a vertical angle on each end. The ends, which are a solid welded frame, covered with heavy sheet-iron, rest against the sides. The sides and ends are held together by the clamping bolts shown. One end of these passes through one side and is bent over, so that when the end with the fly-nut, which drops into a slot on the other side, is loosened to take down the frame, the bolt drops down out of the way but does not become detached. Sides and ends are held to the bottom by a clamping-bolt and wing nut shown between the wheels, and make a tight joint with the bottom by means of a hardwood capping piece fastened to the bottom proper, which projects up between and is surrounded by both ends and sides.

The ends of the frame marked *B* are of wood and are held to the bottom of the frame by means of a hinge which operates in an eye bolt fastened to the bottom. The sides are clamped to the ends, around which they fit closely, by means of levers. To take the frame apart the lever handles are pulled down, the wing-nuts loosened and the sides removed, and finally the ends.

The frame marked *C* is similar in construction to *A*, but with a different clamping arrangement at the ends. Two tapered lugs are riveted

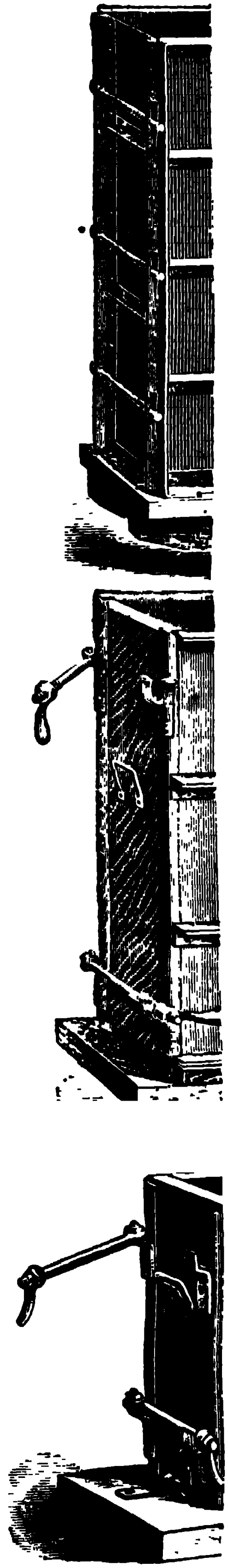


FIG. 72.—Methods of Frame-locking.

to the sides, and the clamping pieces which are forged to fit these are driven down, drawing the two sides together.

The frame marked *D* is similar in construction to *B*, but the bottom is somewhat different. A hardwood strip, *A*, is hinged to the bottom and holds the sides tightly against the top-board, *B*, as shown. A similar piece, *C*, topped with an angle-iron, *D*, holds the ends likewise, at the same time holding down the side-binder, *A*. A latch, *E*, holds the piece *D* in place. It is therefore only necessary to push back the latch *E*, which will allow the side and end binder-pieces, *A* and *C*, to be thrown back. The frame is then taken apart by pulling down the clamping levers *F*, as shown

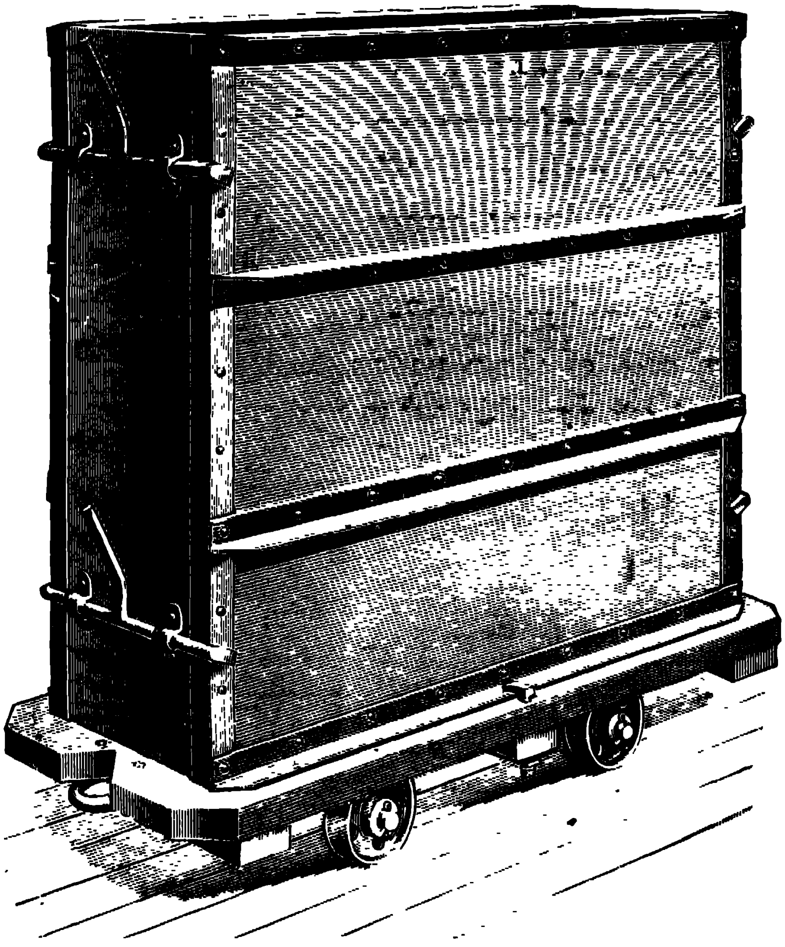


FIG. 73.- Soap-frame.

In the various types of frames illustrated there is shown great variety in methods of fastening. In fastening the sides and ends of the frame shown in Fig. 76, a novel adjustable clamp is used which is shown separately in Fig. 75. This clamp once adjusted to clamp the ends tight, need not be disturbed for a long time.

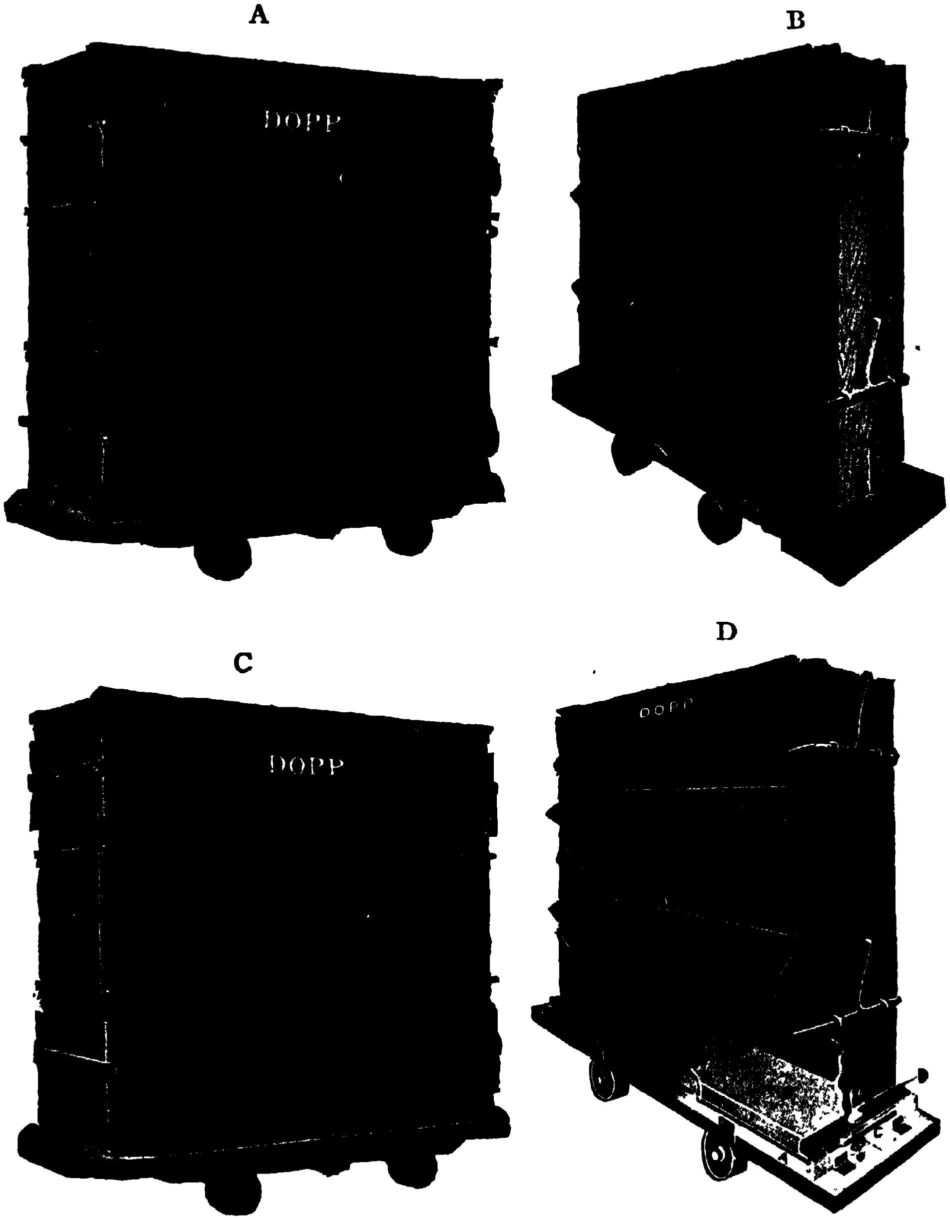


Fig. 74.—Soap-frames.

The eccentrics lock themselves, and cannot open through careless handling. The opening of the clamp permits it to swing free from the sides, allowing ready removal of the different parts.

Under conditions commonly prevailing the filled soap-frame is a cumbersome body to move. A device to facilitate its transport is shown as actually used in Fig. 76, and in its elements in Fig. 77. These plates may be applied to any soap-frame, and in conjunction with the pivot-truck makes a most desirable improvement in the usually cumbersome and laborious method of handling frames.

The method of fastening and the device for moving the frame shown in Figs. 75, 76, and 77 are the subject of letters patent, the object of this invention being, first, to provide a means of fastening the movable upright sides of the soap-frame in a manner which will insure the utmost facility in placing them in their fastened position ready to receive the mixed soap and also to render the removal of the sides (so removed to be used upon another bottom to receive a second supply of soap) performable with ease and despatch; second, to provide a means of moving and guiding the soap-frame (with or without its load), which will be adjustable according to the weight of the apparatus and the strength and height of the person; third, to provide a firm support for the separate distinct pieces comprising the platform of the soap-frame; fourth, to provide a means

Fig. 75.—Adjustable Clamp for Soap-frame.

of holding the platform in rigid contiguity with the above-mentioned four upright sides. In stripping a frame set up and fastened as described, the first step being to turn down each of the three clamps on both sides of the bottom; next to turn back and release the end-fastenings; thirdly, to detach the sides and to stand them in position to use again, and lastly, to detach the ends. The pivot truck is now brought into use to easily and quickly move the stripped frame of soap.

In the frame shown in Fig. 78 is displayed a marked departure from the conventional style of frame construction. The bottom consists of a wood base about $1\frac{3}{8}$ inches thick, to which is attached

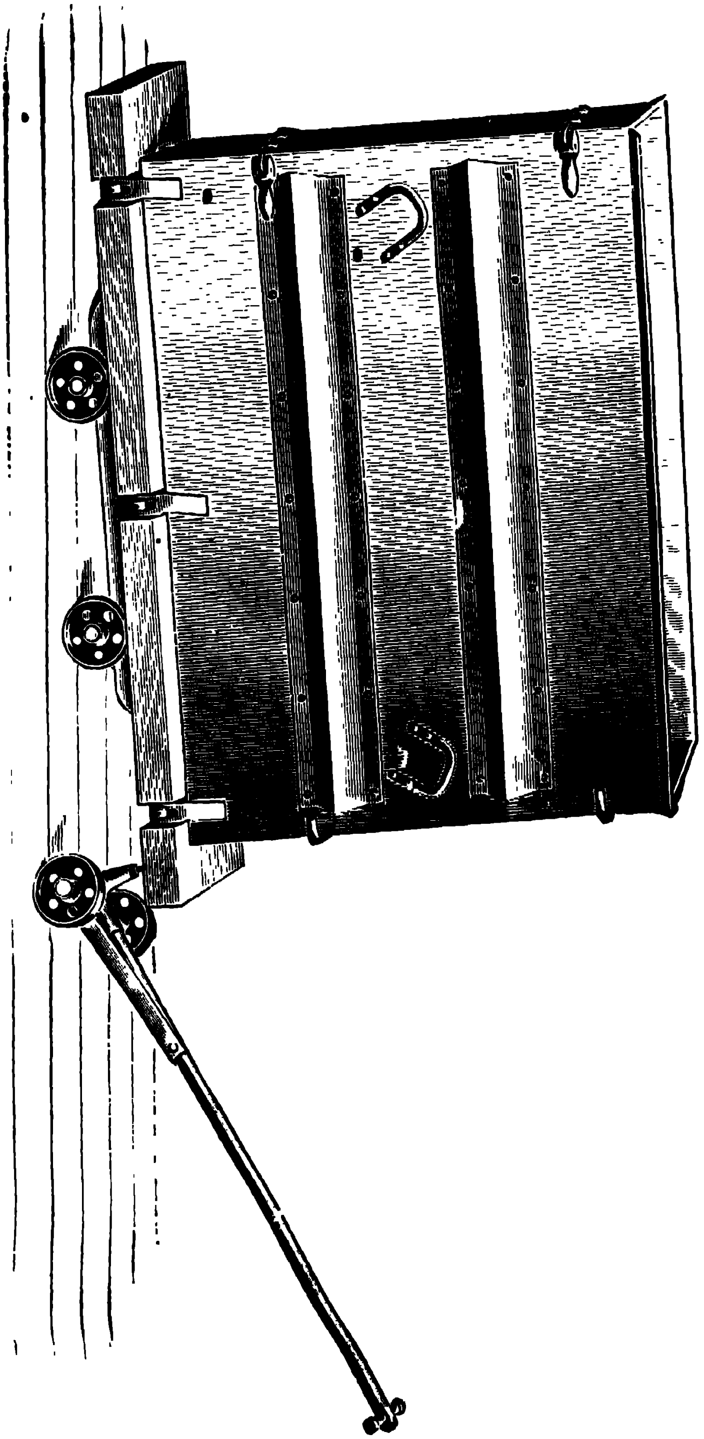


Fig. 76.—Device for Moving Frame, as applied.

a single sheet of steel with a hooked flange turned up at the four edges. The sides are made of sheet steel, strengthened as shown boilings each filled frame should be marked with a card bearing its number, quality, if necessary, and the date of framing so that some record of it is possible. The frame-room should be cool and well-ventilated, in many instances the cellar being used for this purpose.

The period during which soap stands in frame is variable and is determined by the character of the soap, dimensions of the frame,

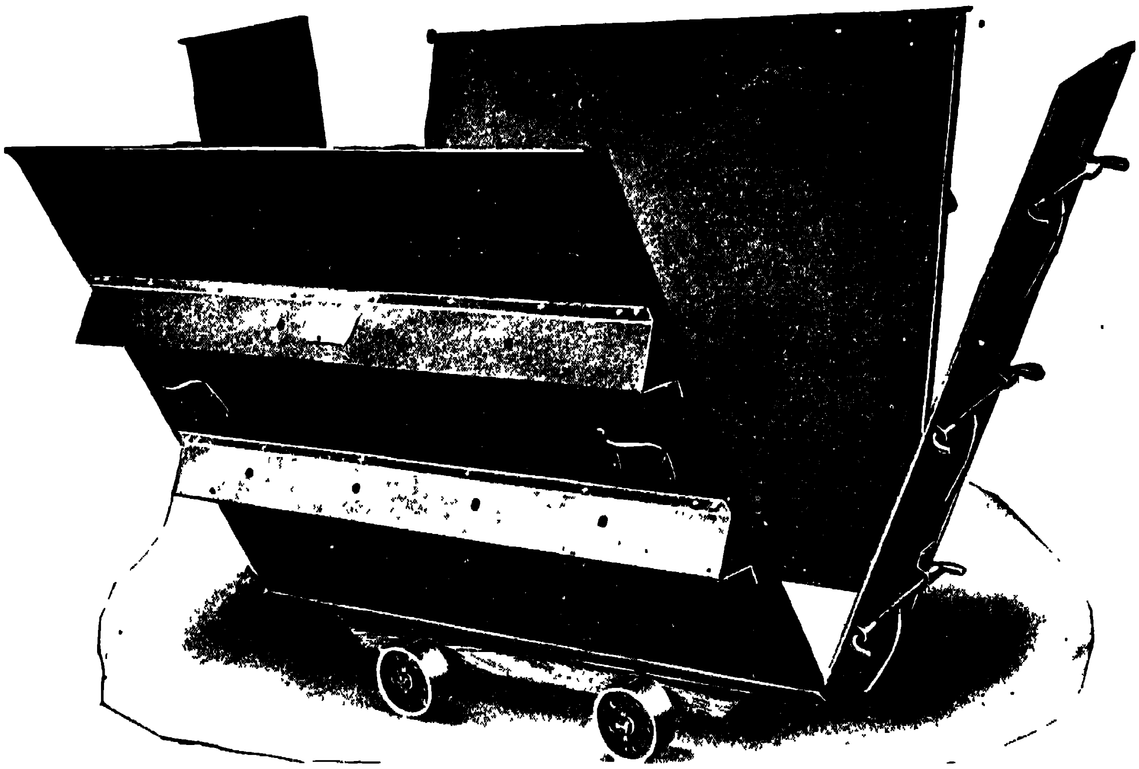


Fig. 78.—Soap-frame.

temperature and season and the demand for the product. Three days is an average period, although in warm weather, five days may be required for cooling. In stripping, the frames are unlocked, and the sides first removed and lastly the ends. Often trouble is experienced with soap adhering to the sides which a wedge will not overcome. Water or brine may be poured into the initial crevice and allowed to work down. The wire is the last resort.

The Slabber.—Mechanical slabbing devices contain the germ of the primitive system wherein a wire is drawn, in parallel lines of uniform distance apart, through the frame of soap. This system is yet followed in many factories and exists in various stages of development throughout the industry. Where a large number of brands of various sizes are cut, and from the same grade of soap, a hand slabber, Fig. 81, represents the greatest practicable economy. An advance in efficiency of operation is indicated by the use of slabbers

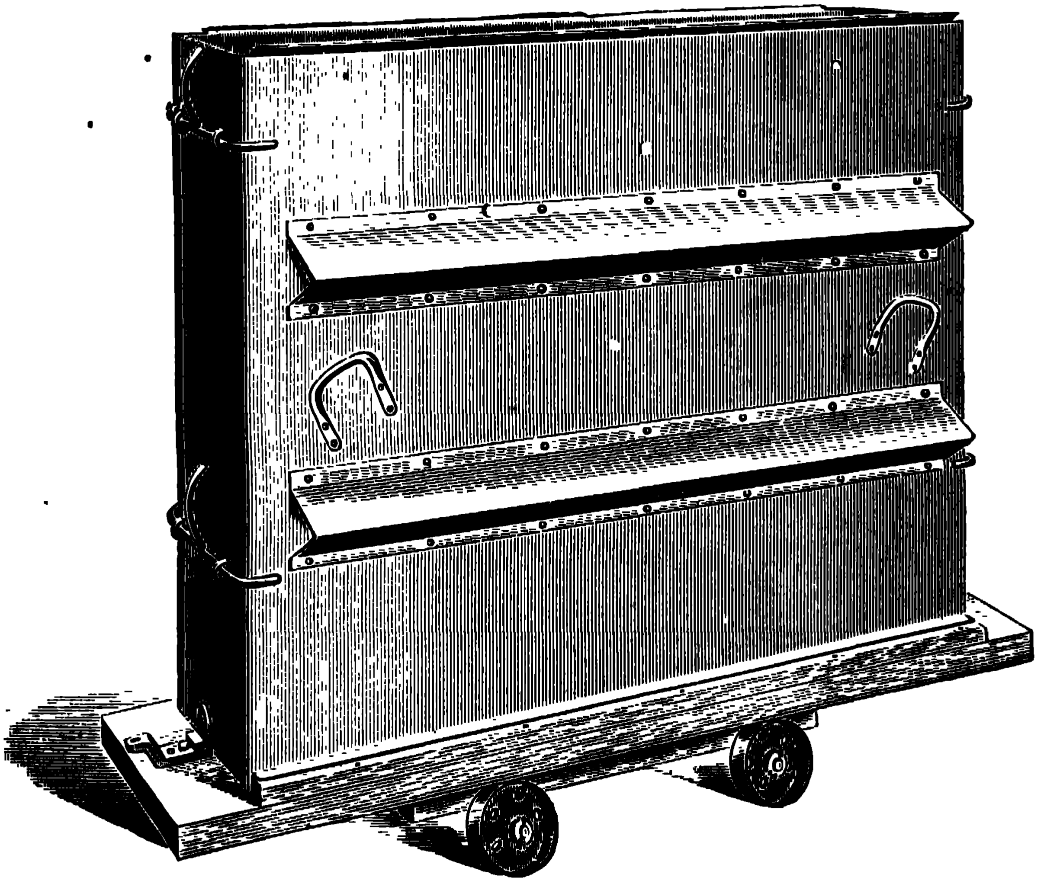


FIG. 79.—Soap-frame.

of the type shown in Figs. 82 and 83, their method of operation being evident from the illustrations. In the Ralston slabber, Fig. 82, the cutting head is stationary, the soap being impelled through it. In the Champion slabber, Fig. 83, the cutting head is movable and is drawn through the soap.

In the belt-driven slabber shown in Fig. 84, the cutting head is movable. This slabber is arranged with two open ends, a frame of soap is rolled in the front entrance and the gate or stop is closed up against the soap; the clutch shifter is moved, which starts the sliding head, carrying the wires through the soap. When the wires have passed through, the clutch is automatically shifted out of gear; the operators now draw out the slabbed frame of soap from the rear of the machine. When this is done the wire-head is reversed and it goes back to its starting point again, ready for a new frame.

It takes about a minute to run a frame of soap into the machine, close the gate, run the wires through and remove the frame. Considering the time it takes to handle the soap about the floor, a fair output might be considered at 150 frames a day.

This slabber is constructed entirely of iron, is rapid of operation, and requires a minimum of labor. Where volume of business warrants, its use represents the highest efficiency of operation.

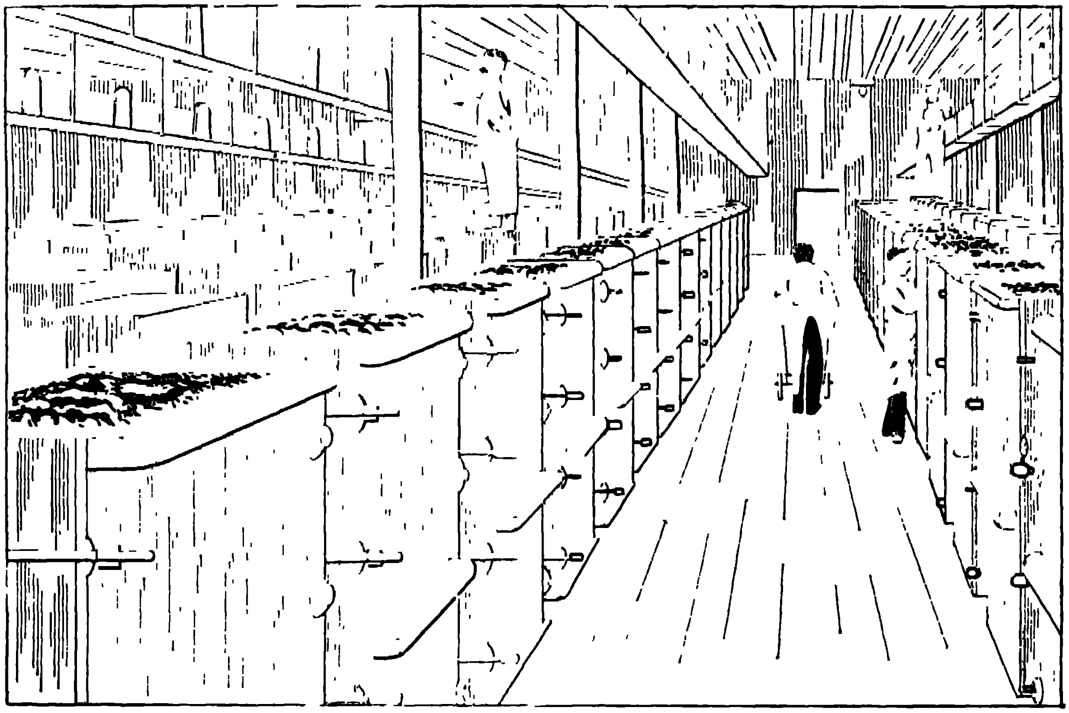


FIG. 80.—Frame-room, Frames Filled by Trough System.

A belt driven slabber of cheaper construction than the type shown in Fig. 84, but of equally efficient operation, is shown in Fig. 85. The mode of operation is identical. The use of wood permits a selling-price within the reach of smaller factories. The frame as shown is substantially braced and bolted.

The Cutting-table. The purpose of the cutting-table is to resolve the slab, cut as a rule to a thickness that corresponds to the width of the cake, into the maximum number of cakes with the least amount of scrap, by means of two cutting movements at right angles to each other. They may be operated either by hand, as in the type shown in Figs. 87, 88, and 89, or by steam-power, as shown in Figs. 90, 91, and 93. The cutting heads of both types of machines are replaceable, whereby the slab may be reduced to cakes of varying

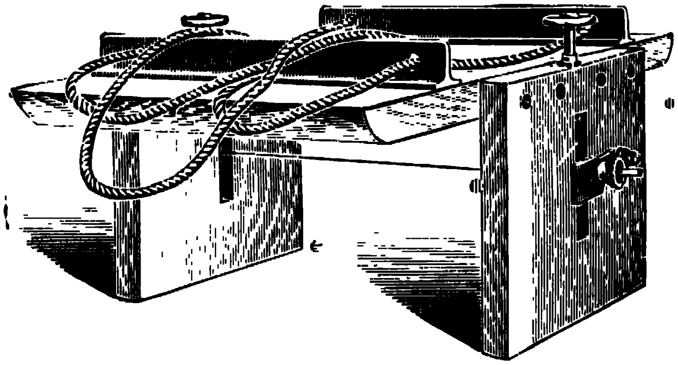


FIG. 81. —Hand Soap-slabber.

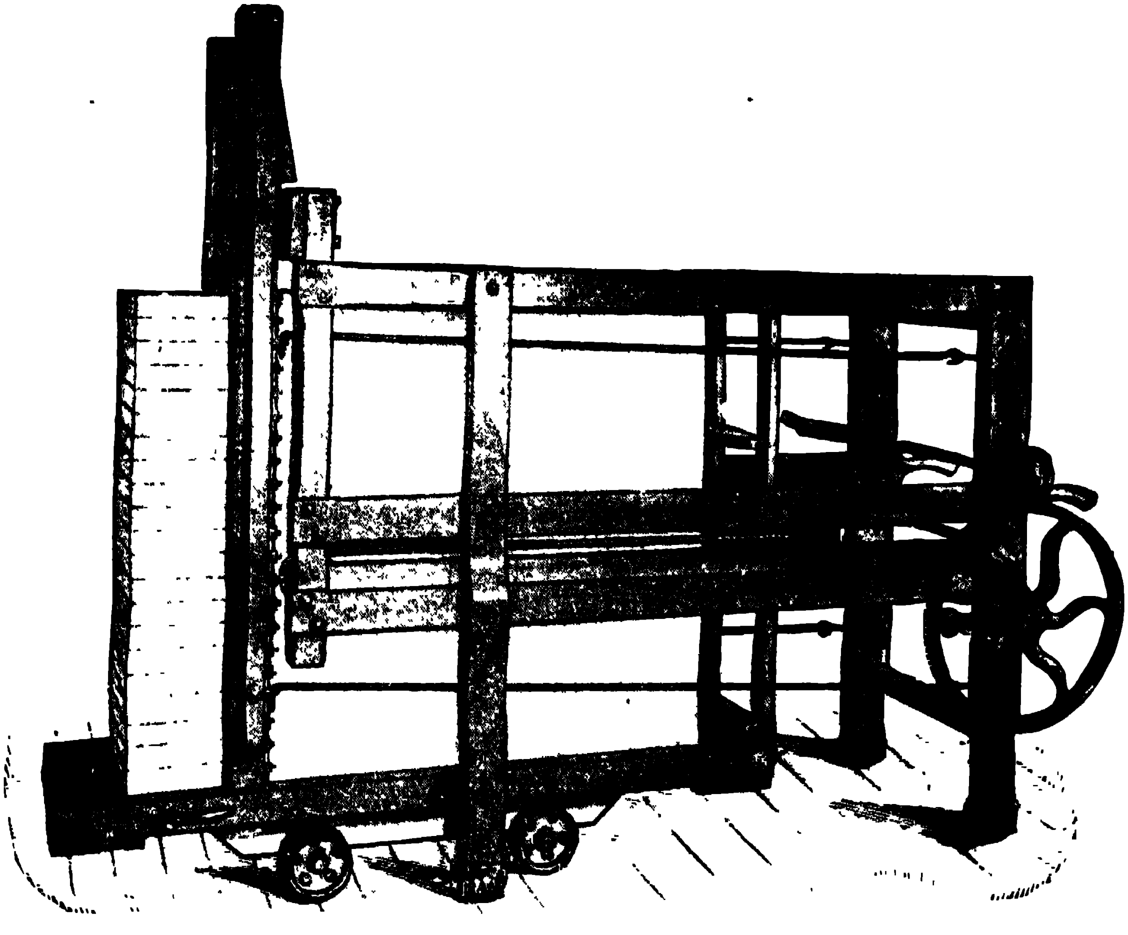


FIG. 82.—Ralston Soap-slabber.

dimensions according to the distance separating the wires in the different cutting heads.

The mode of operation of hand cutting-tables is clearly shown in the diagram, Fig. 86. In its operation the two attendants in turn change their relative position to lift the slab onto the table; the first turns the handle that runs the soap through the first set of wires; then the second operator runs the soap through the second set of wires onto the racks by changing his position to the end of the machine; the two men then lift off the filled racks and proceed as before.

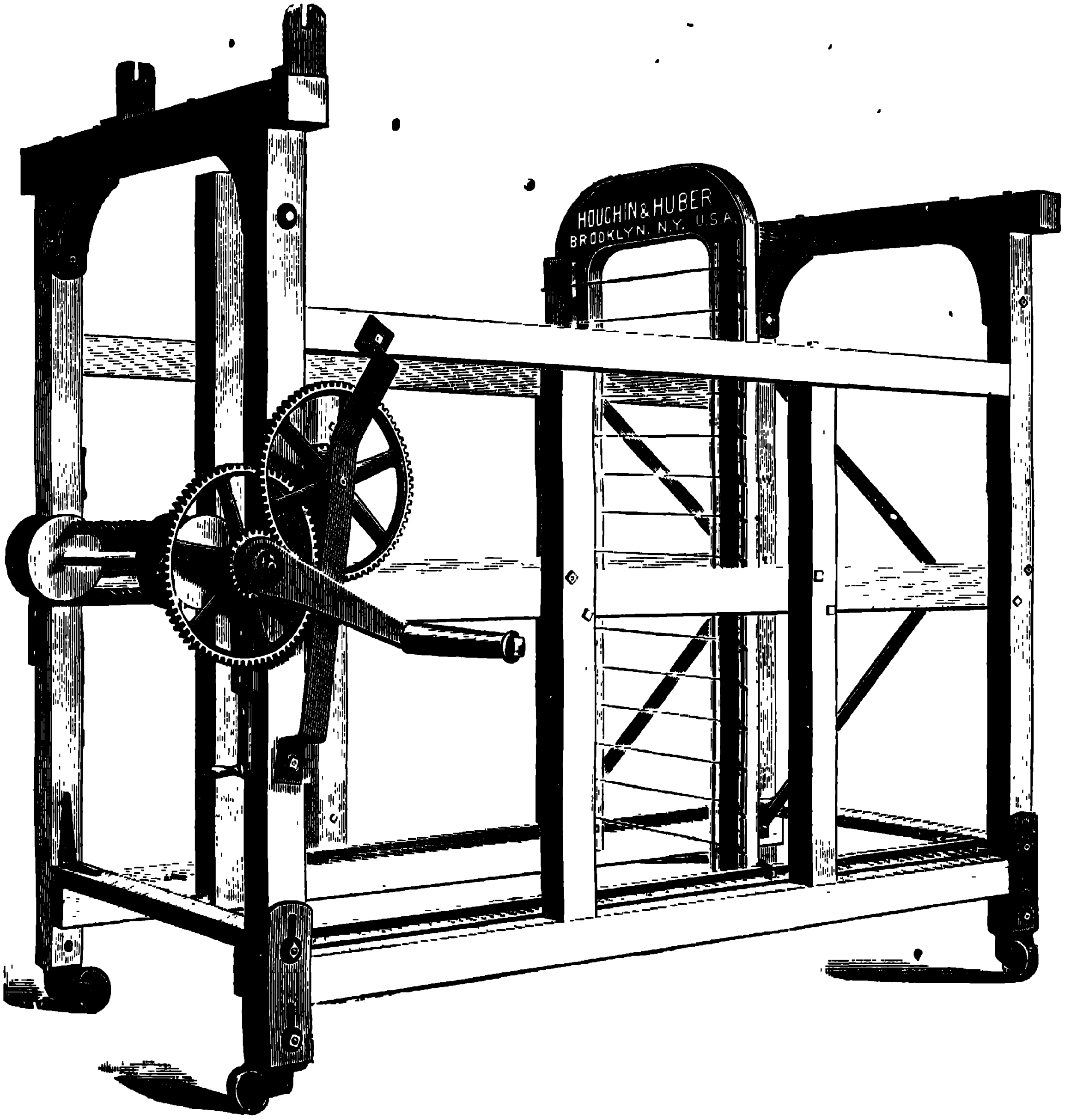


FIG. 83.—Champion Soap-slabber.

In the power cutting-tables the operation is continuous and automatic; the slab of soap is placed on the first table, Figs. 90, 91, and 93, and the machine started. In passing through the first head, the sides of the slab are trimmed and the remainder cut in longitudinal slabs, which in turn on the second table are subjected to a movement at right angles, whereby they are forced through the second head and resolved into cakes of the desired dimensions. One or two racks may be filled. Two racks may be filled at once by both hand- and steam-driven tables by the use of "fingers" on the second driving-head, and by projections on the rack support, whereby the movement of alternate strips is interrupted so that two racks are filled and soap on them uniformly spaced. The speed of automatic tables is limited only by the ability of the operators to supply slabbed soap and to remove the same on racks. As rapidly as filled the racks are transferred to suitable trucks; a truck having received its quota of racks is ready to be drawn into the drying-room, where the individual bar is left to assume a consistency best suited for pressing. The methods employed for handling soap at this stage vary greatly with each factory.

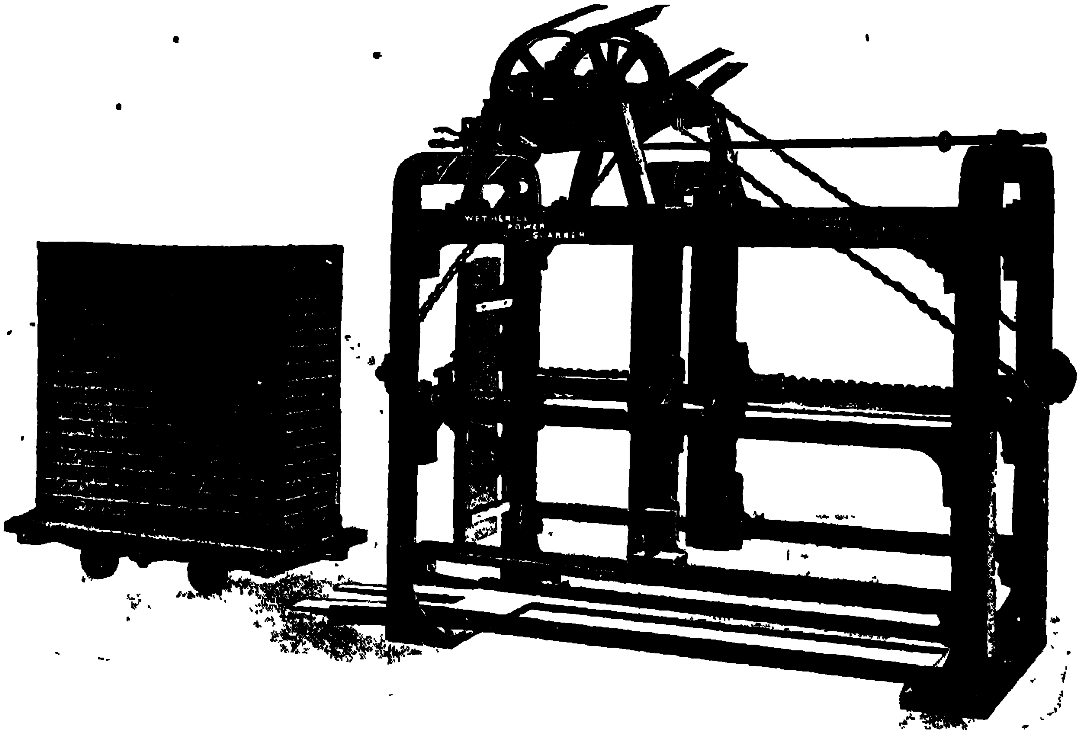


FIG. 84.—Wetherill Power-slabber.

The cutting-table shown in Fig. 90 is semi automatic in operation. By moving a lever the pushing-block of the first table forces the slab placed thereon through the first cutting-head, and then returns to the starting-point, stopping automatically. The corresponding movement is effected in like manner on the second table to which is attached the racking and spreading device.

A very compact and easily operated hand cutting-table is shown in Fig. 87. By means of the crank in front of the table the slab is pushed through the first set of wires; by means of the crank at the side of the table the slab is then pushed through the long header upon the other side of the table. Here the soap may be removed and racked by hand. By attaching a spreader-device and a rack-support to this side of the table, the soap may be pushed upon the racks by means of the slab of soap behind, and the operator by merely lifting one end of the rack filled with soap and pulling it with sliding motion up the inclines, effects a uniform separation of the soap throughout the length of the rack. By use of the hand cutting-table shown in Fig. 88, spreading of the soap on a pair of racks is effected by the same movement of the crank that effects the second cutting of the slab.

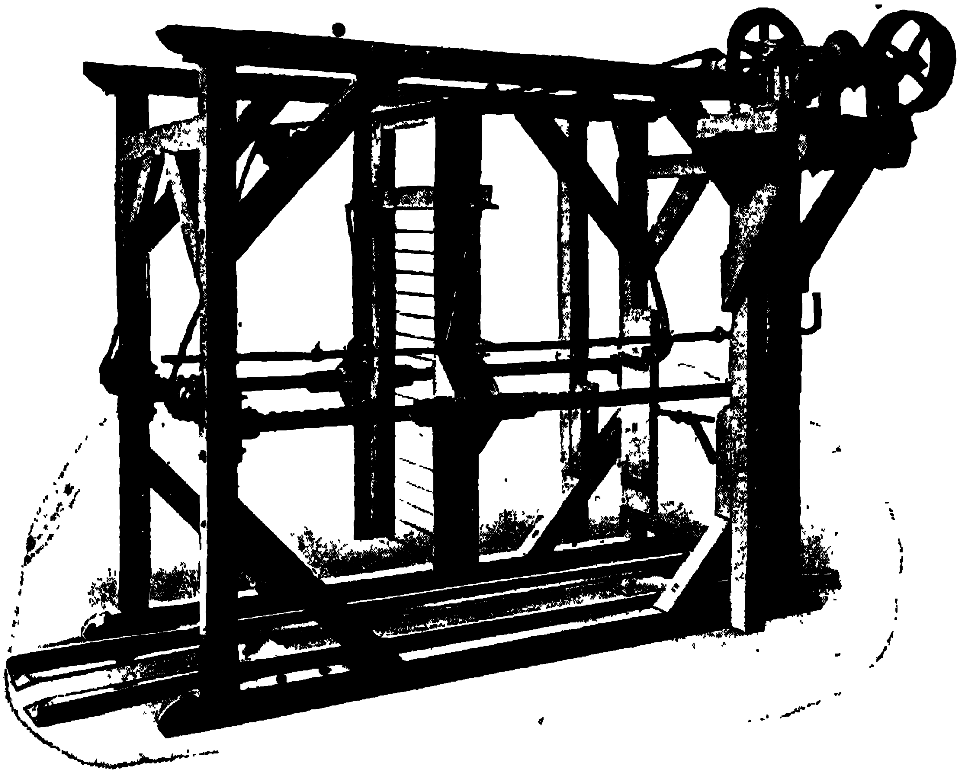


FIG. 85.—Automatic Power-slabber.

A cutting-table constructed entirely of heavily enamelled iron is shown in Fig. 89. The pusher-blocks *A* and *B* are connected by chain and sprocket to shafts on which are the hand-wheels, *C* and *D*, by means of which the operators effect the movement of the slab.

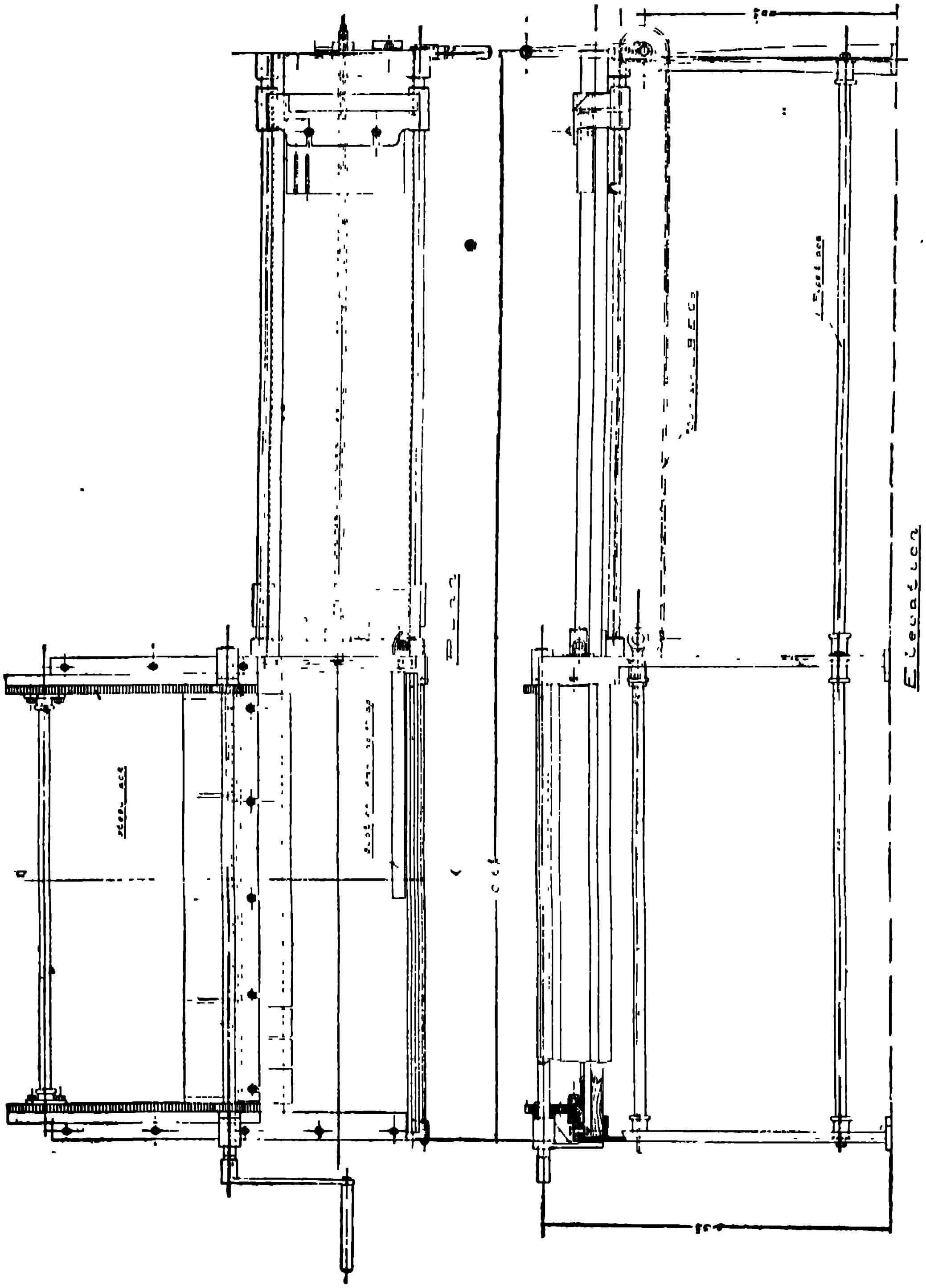


FIG. 86.—Diagram of Hand Cutting-table.

The Wetherill automatic cutting-table, Fig. 91, consists of a cast-iron frame supporting two tables, each having a pusher to force the slab of soap through the cutting-heads. The first pusher is guided on two steel circular guides and is driven by means of two link chains passing over the guides; the second pusher runs on cast-iron guides machined and scraped to fit. This is driven by means of a machine-cut rack and pinion. These pushers run automatically, starting, stopping, and remaining idle at the proper intervals. These movements are performed by one master-wheel, Fig. 92, which does the work without the use of cams, levers, springs, ropes, or drums.

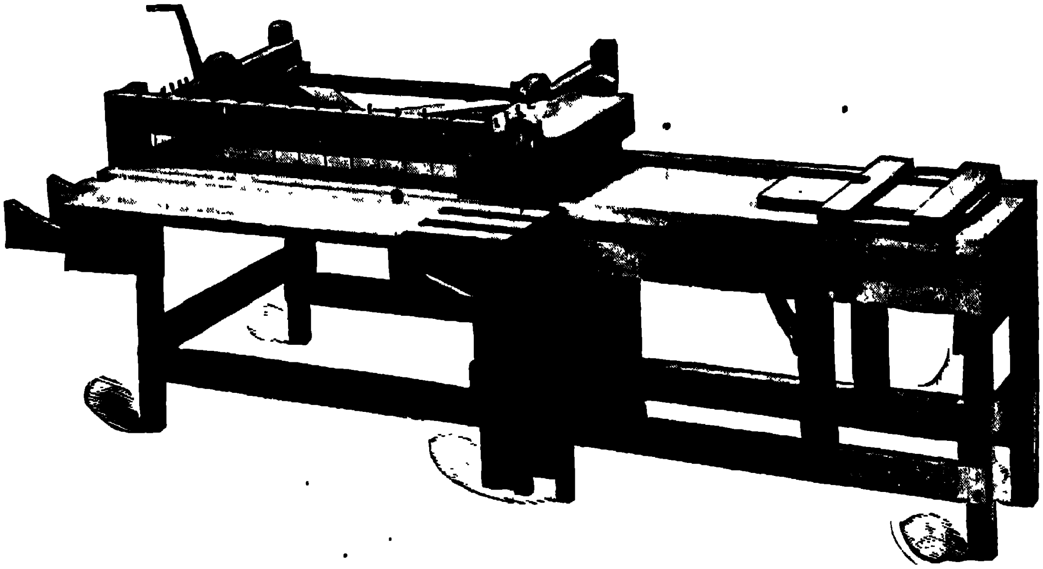


FIG. 87.—Ralston Cutting-table, with Hand-spreader.

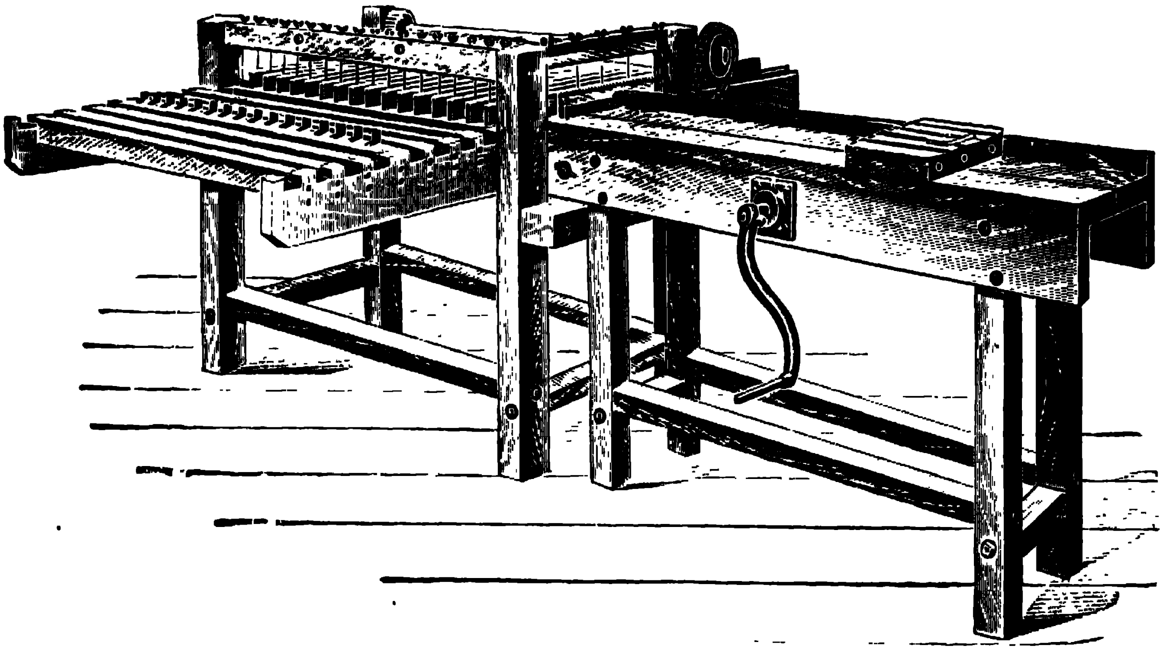


FIG. 88.—Cutting-table, with Automatic Spreader.

The cutting-wires are held in movable finished steel frames which can be slid out and other frames with different spacing of wires inserted. The wire-holders are small hooks with a thread

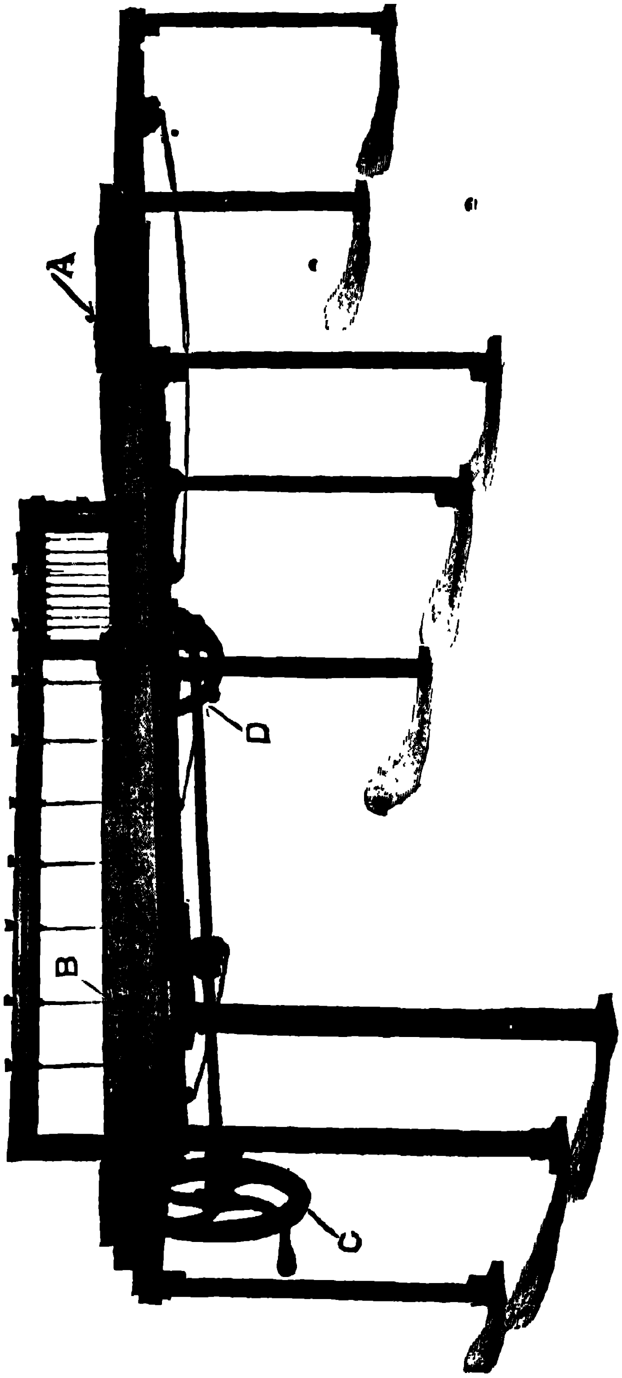


FIG. 89.—Iron Cutting-table.

and thumb-nut. The wires are twisted around the hook and inserted into the frames. Hard-wood blocks in the frames separate the wires the proper distance. Three strong boys are sufficient to operate the cutting-table. Two are stationed at the long table and trans-

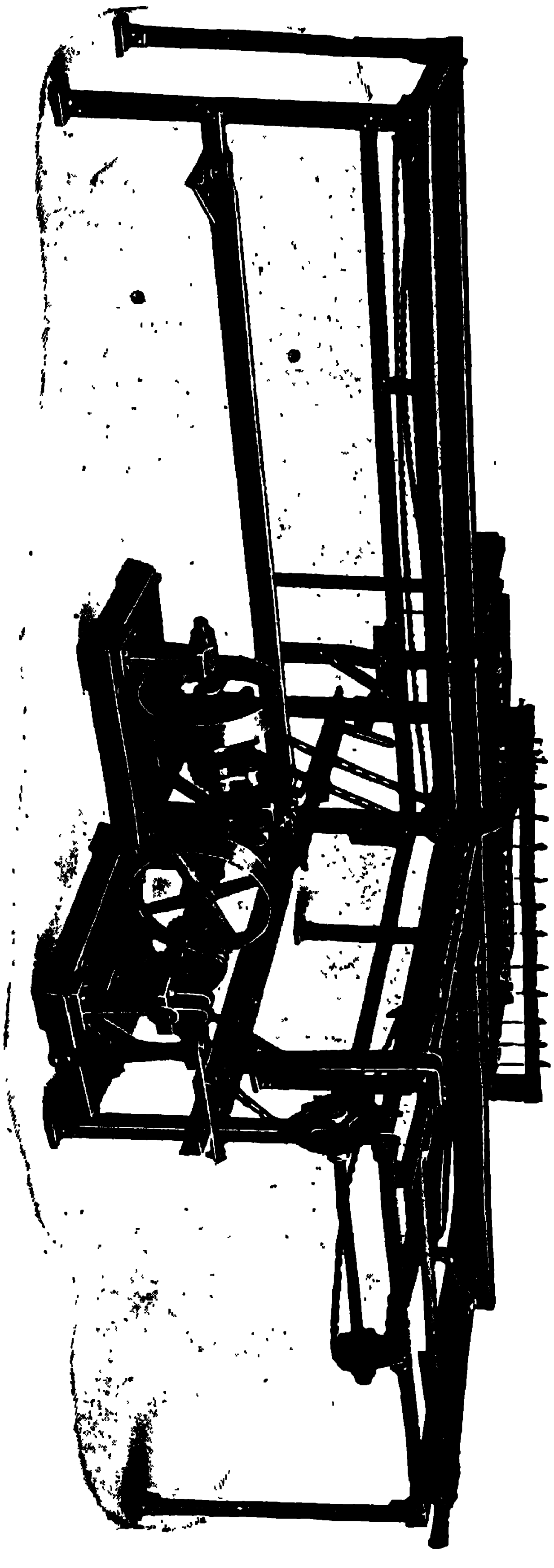


FIG. 90.—Semi-automatic Cutting-table.

fer the slabs from the frame to the table, while the third operator helps to lift off the racks of soap when filled and to place empty racks in their proper position to be filled.

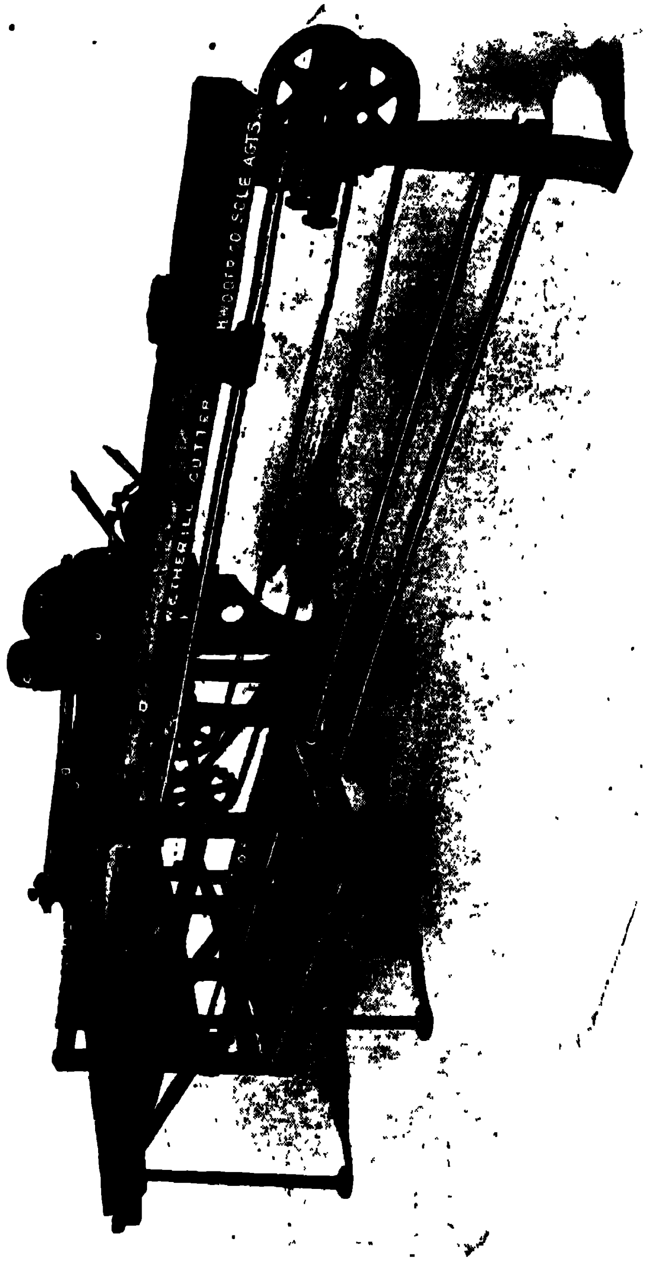


FIG. 91.—Wetherill Automatic Cutting-table.

The construction and operating mechanism of the Detroit cutting-table are shown in Fig. 93.

Scrap.—The percentage of scrap obtained on slabbing and cutting depends upon absence of air-holes, foreign matter, etc., in good soap, the degree of cracking in soap crutched and filled too hot, the agreement of bar and frame dimensions, and the shape of the bar—more scrap being made with an oval bar than with one of rectangular cross-section. It varies from 12 to 25 per cent of the total weight of soap in the frame. It should be endeavored to reduce the percentage to as low a figure as possible. The disposition of scrap is determined by a variety of circumstances; it may be either added

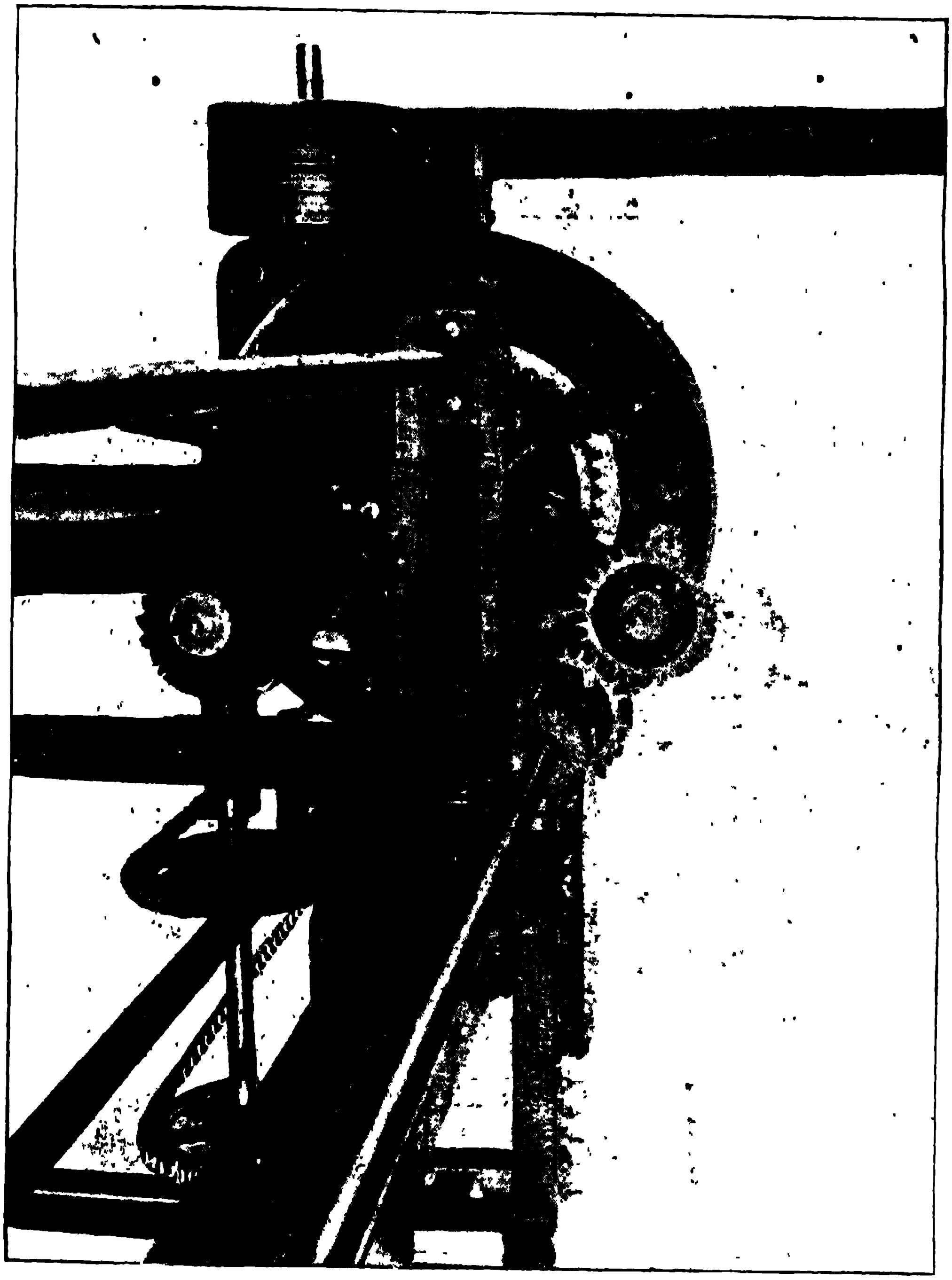


FIG. 92.—Master-wheel of Cutting-table.

to the kettle on the strengthening change, or remelted and framed directly, or the remelted soap mixed in varying amounts with fresh soap in the crutcher. Whatever procedure is employed depends upon the volume of scrap, the cost of filling, and, what is most important, the appearance desired in the finished product. Where utmost clearness of texture and brightness of color are desired in the finished product, where soap is made on a comparatively small scale, the scrap is returned to the kettle. With a large volume of scrap and less attention paid to the appearance of the finished product as stated, the scrap is remelted and framed directly or mixed with fresh soap in the operation of crutching.

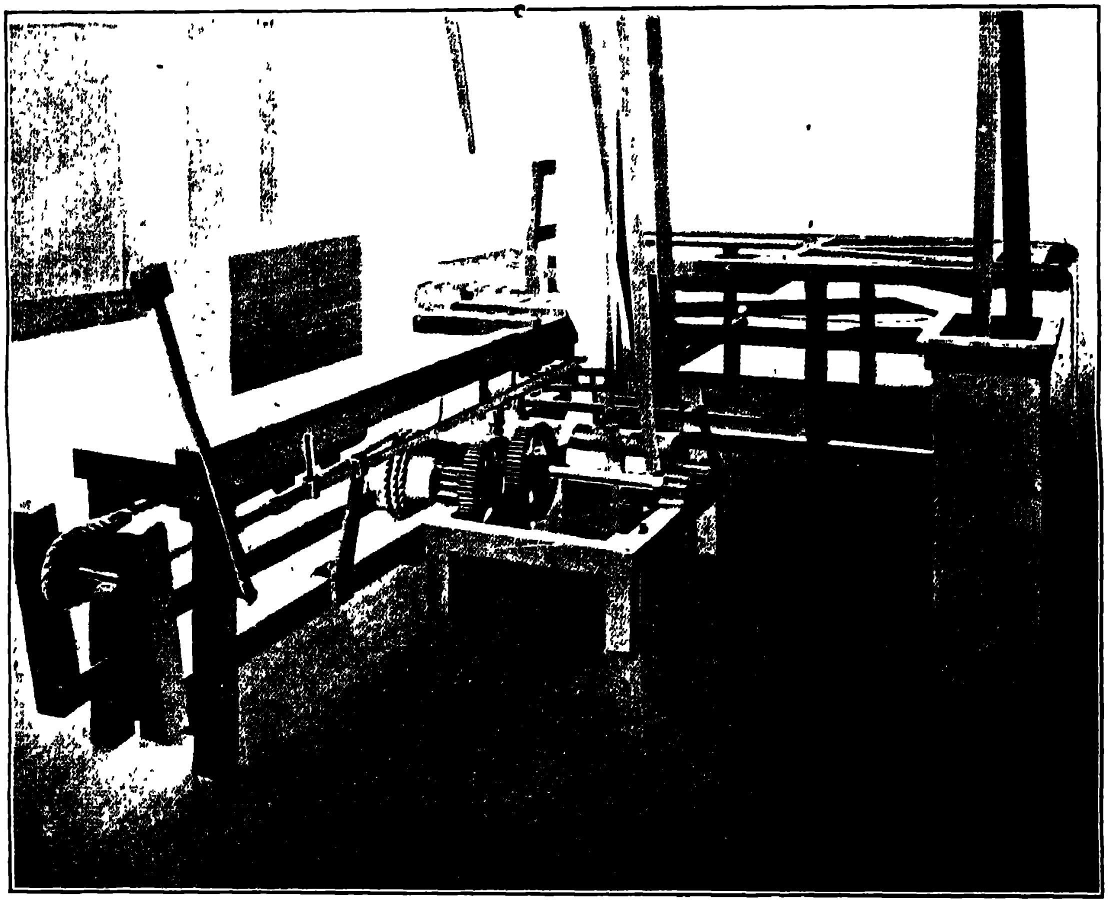


FIG. 93.—Detroit Cutting-table.

The Remelter.—In factories of small size, where the volume of business does not warrant the installation of a separate remelter, the remelter is very conveniently combined with the crutcher by providing any type of crutcher with a steam-jacket. The essential features of a remelter are shown in the conventional form represented in Fig. 94.

The machine is composed of a sheet iron tank having a system of steam-pipes arranged inside, and a grating for the soap to rest upon. The steam pipes are arranged so that circulation of steam takes place throughout the complete nest, and an exhaust is arranged at the bottom of the tank. In addition to this a pipe is arranged for admitting live steam directly into the tank. When the remelter has been filled with scrap soap live steam should be admitted to the coil for about ten minutes, and at the same time steam should be admitted to the nest of pipes. At the end of about ten minutes the live steam can be shut off and the remelted soap let out at the discharge-gate in the bottom of the tank directly into the crutcher, and after mixing in this machine it is ready for the frame. Open or live steam should be let in from time to time for about ten minutes.

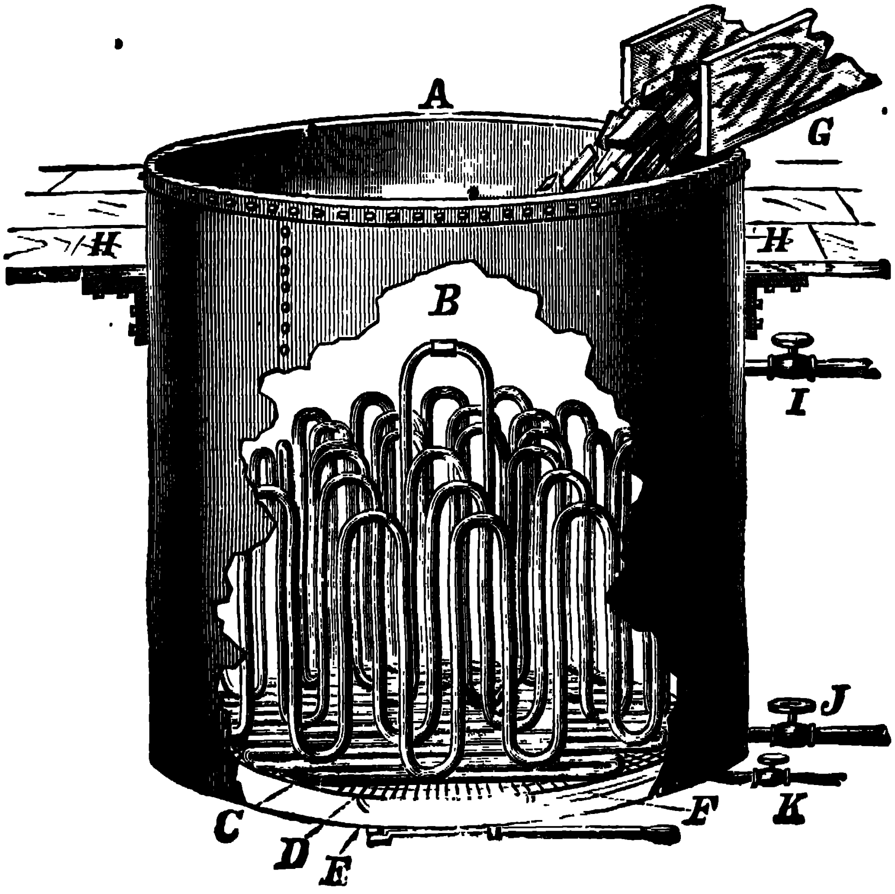


FIG. 94.—Remelter.

To give the proper results the remelter should have its steam-pipes so arranged that each has an independent supply of steam and discharge for condensed water. It should heat the soap evenly, melt it, and as fast as it is melted automatically, remove the melted soap and replace it with fresh scrap. It must also be rapid in its operation. The remelting-tank proper is insulated by asbestos covering and is surmounted by a strongly braced wood case extending where

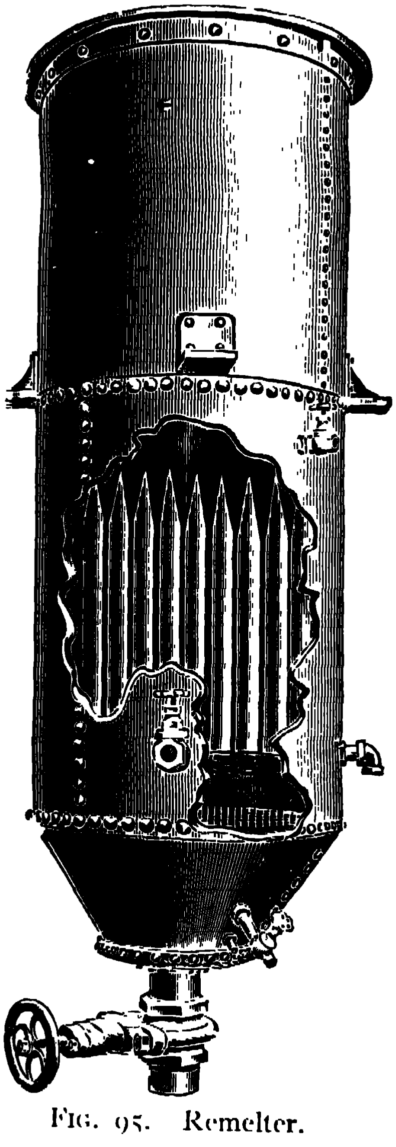


FIG. 95. Remelter.

warranted to any convenient height and designed for the storage of scrap, to which it may be conveyed by hand or by an automatic scrap-elevator of the belt type. Where the volume of business warrants, a separate remelter is used for each grade of filled soap. As the soap melts and is removed, the charge descends by gravity and the operation of remelting and crutching can be made continuous. With this system one crutcher is used for two remelters. The texture of remelted soap differs from that of fresh soap in being less bright and clear as a result of less complete fusion. Fresh perfume and 10–15 pounds 36° Bé. soda-ash solution may be added to each crutcher-charge. By mixing varying amounts of remelted with fresh soap the appearance of the remelted product is greatly improved. In filling, allowance must be made for that already present in the melted scrap.

A remelter of improved mechanical construction is shown in Fig. 95. The heating device is essentially that of a steam-radiator, providing for the continuous presence of live steam in the heating-coils and for the removal of water of condensation as fast as formed without first traversing the entire length of the heating-coils. At the point of the cylindrical tank where it begins to dish is located the steam-chamber, supporting individual radiating pipes between which and through suitable openings in the steam-chamber the soap as it melts descends. Below the steam-chamber is placed a sieve, above which is a pipe for the injection of live steam, and below which in the dish is a closed steam-coil. The remelter is provided with a gate-valve of ample dimensions. To start the remelter, steam is admitted into the closed coil located in the dish. Where the moisture-content of the soap is such as to warrant the use of live steam, this is provided for by the open coil located above the sieve. The high efficiency of the heating system provides for a continuous flow of remelted soap with a minimum expenditure of steam.

Drying of Soap.– Freshly cut soap is soft, sticky, and opaque, and, if properly crutched, should be homogeneous. It contains from 30 to 35 per cent of water, depending upon the manner in which the soap was finished, and upon the nature of the addition during crutching. In calculation, 100 parts of neutral glycerides are considered to yield 150 parts of finished soap. An analysis of a pure curd soap at this stage presents the following data: Fat anhydrides, 61.80 per cent; combined alkali, Na_2O, 7.21 per cent; water, 30.99 per cent.

The presence of a greater amount of water than is here shown would tend to indicate an intentional addition, its incorporation being made possible without excessive softening by the use of soda-ash and sodium silicate.

Previous to the introduction of the rapid-drying apparatus the moisture in the exterior parts of the bar was allowed to evaporate spontaneously. By this method the drying was an extremely slow process, and also very unsatisfactory in that the weather was the arbiter of the output. A stove in a closed room was a great improvement. This primitive method, with its manifest disadvantages, was succeeded by a system of hot-air circulation by natural draft. Steam heat by simple radiation from pipes was also employed, and with the introduction of forced draft the elements of the system of drying at present in use were established.

The purpose of the drying-room is to hasten the evaporation of water from the surface of the bar so that there may be formed a thin crust of comparatively hard soap, which serves to retard further evaporation from the interior, and which allows the bar to be pressed and stamped without the soap adhering to the dies. Without the formation of this skin of firm soap, the latter process is impracticable. On cutting a bar of soap into halves, this superficial drying becomes plainly evident. The soap when removed from the drying-room, and after pressing, has a smooth, glossy, and translucent surface, which condition is in marked contrast to that observed at the cutting-table. During the drying process from 3 to 5 per cent of water has been expelled entirely from the surface of the bar, while the interior contains the amount of water originally present, viz., 30 to 35 per cent.

The appearance of soap is affected by the evaporation of water from within the mass, and by the absorption of water from without. Drying ensues at all times, but diminishes as the dew-point is approached when it reaches zero. Moisture condenses upon the surface or is absorbed at a rate increasing as the dew-point is reached, where the tendency for any soap is at its maximum. It varies with different soap according to its stock and method of manufacture. It is greatest with olein stock, of which cottonseed-oil is the chief representative, and rosin, and greater with soap made by the boiled than by the cold process. The "sweating" of soap is directly as its solubility, and is least with soap made from firm stock.

In the drying process air fulfils two functions: it carries to the moist soap the heat necessary for evaporation, and serves as a vehicle for the removal of moisture. The capacity of air for heat is very small, its specific heat being but .238, with water as 1. Its capacity for moisture depends directly upon its temperature and its relation to the dew-point, naturally diminishing as the point of saturation is reached. With rise of temperature the capacity of air for moisture greatly increases. It is estimated that air at 72° Fahr. has a threefold greater capacity for moisture than the same volume at 42° Fahr.; at 132° Fahr its capacity has increased 20 times, while at 172° Fahr. its capacity is more than 80 times as great. Increase of temperature thus means the more rapid formation of vapor, with a much greater increase in the capacity of air for absorbing it. Add to this the rapid circulation of air, presenting to the moist surface at every instant an atmosphere greedy for moisture, and the great convenience and efficiency of artificial drying become evident. The essential requirements of the heating and ventilating apparatus of the drying room are that it should provide a large volume of air at the requisite temperature and maintained in rapid circulation. A comparatively low temperature, 80 100° Fahr., is productive of the best results. A temperature in excess of 100° Fahr., or air maintained at this temperature for any considerable period, is not only unnecessary but undesirable. At this temperature, if allowed to remain too long in the drying room, the soap undergoes an appreciable softening with the development of more or less discoloration. The temperature at which soap will melt depends primarily upon the nature of the stock from which it is made and the proportion of water with which it is associated. In the drying of green soap it is desirable that the currents of warm air should circulate lengthwise of the bar, in order that the largest extent of evaporative surface may be exposed, and the drying process thus hastened.

Aside from the processes in the kettle, there is no stage in the manufacture of settled soap that requires greater care in its operation than the treatment received in the drying room. Improperly dried soap, while it may cause no great trouble in the press, is subject to rapid deterioration in appearance, and the influence of this one factor on its ultimate distribution demands that its final treatment in the factory be the subject of the closest attention of the soap-maker.

Drying-rocm.—The mechanics of the modern soap-drying room represent more than the translation of a similar process employed in other departments of industry than it does a natural evolution from previous efforts in this particular field. The use of the centrifugal fan in the production of artificial draft dates from the sixteenth century, but it was not until Stevens' experiments in the early part of the last century that the devices for artificial draft resolved themselves into the two systems of ventilation we know to-day, viz., the plenum and vacuum, or, respectively, forced and induced draft. These found their first application in the firing of boilers. The use of centrifugal fans in this connection, however, languished at first, but with the growing demand for increased

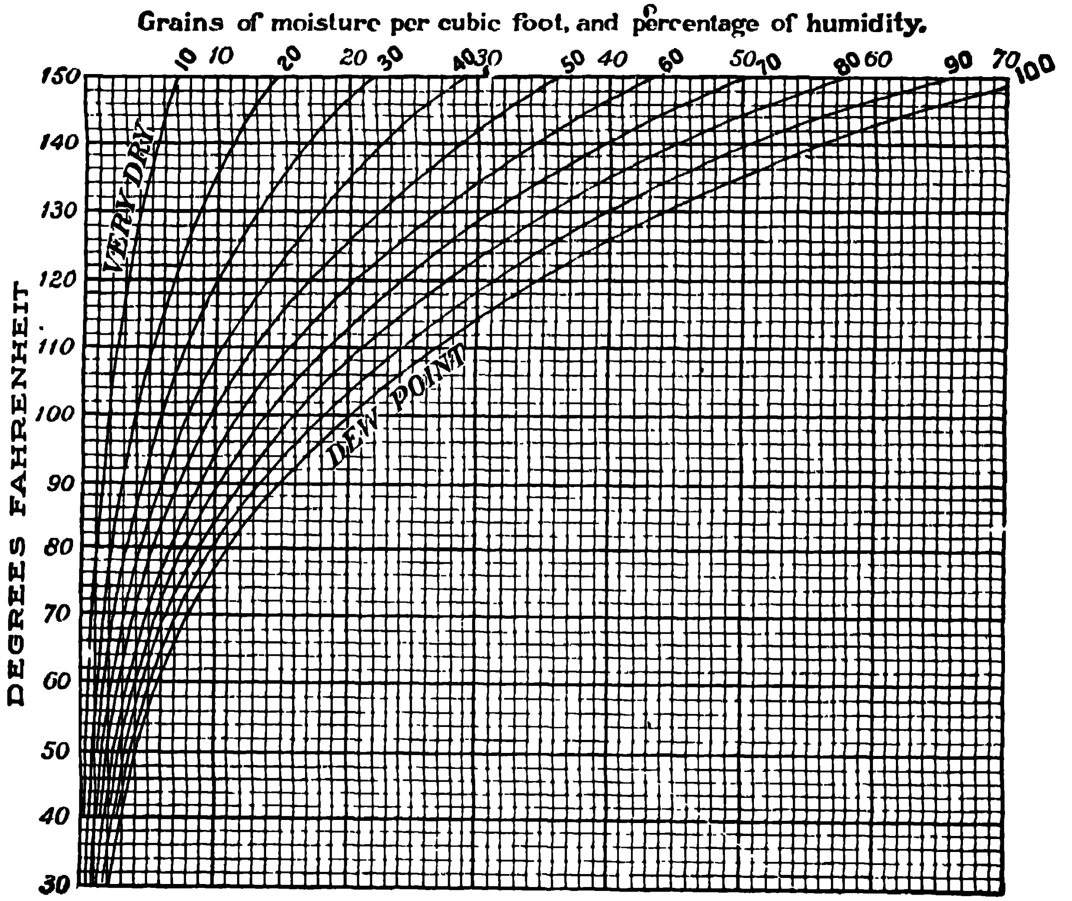


FIG. 96. Hygrometric Chart.

As heat rarefies the air it increases its capacity for carrying off moisture, as represented by the curved lines of the chart. The curved lines represent 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 per cent humidity or moisture, 100 per cent being the dew-point.

engine speed and higher steam pressure their employment as a substitute for natural draft in the combustion of fuel has become almost universal. The application of the improved fans in the heating and ventilation of large buildings was a natural step. Considered more intimately in connection with the drying of soap, venilating-fans may be divided into two general classes, viz., the centrifugal fan or blower and the propeller or disc fan. The former is more generally confined to ventilation by forced draft and is designed primarily for removing air under pressure. Fans of the disc type are not adapted for plenum ventilation. They find an extensive use and are very satisfactory for moving air under slight resistance.

Both systems of ventilation, the plenum and vacuum, are employed in the drying of soap. The equipment of a drying-room under the plenum system comprises, as a rule, a centrifugal blower operated either by belt from shafting or by direct- or belt-connected engine and a sectional heater, which consists of steam-pipes enclosed in a sheet-iron case which communicates with the discharge of the fan-case. Air may either be drawn through the heater and discharged at the desired temperature into the drying-room, or the heater may be interposed and the air forced into the drying-room directly from the heater. As the results produced are the same in both cases, convenience of application will determine the arrangement. With forced draft the drying room is so constructed that heated air enters at one end and leaves at the other, while freshly cut soap is introduced from the side, and as the drying progresses it is drawn towards the efflux of heated air and is finally removed at the opposite side. The heater and fan may be placed at opposite ends of the room and the fan employed to exhaust the warm and moisture-laden air. With this arrangement we have an example of the vacuum system, or drying by induced draft. The use of a fan of the centrifugal type is now not desirable. The cheaper and simpler disc fan set in the framework of the wall is, with this arrangement for the drying of soap, equally efficient and satisfactory. The hot-blast drying apparatus, although compact, occupies valuable space. The exhaust-steam connections with the necessary insulation are simple and easily made.

By distributing the pipes of the sectional heater throughout the drying-room in rows parallel to the trucks of soap, and under openings immediately above for the admission of cold air, not only is greater uniformity of the drying process obtained, but the use of the cheaper disc fan, which is admirably adapted for this type of ventilation by exhaustion, is permitted. Exhaust-steam connections are made through the floor, and by means of suitably placed valves steam may be cut off from any section, thus varying the capacity of the drying-room at will. This type of drying-room equipment is in use in many of the largest soap-manufactories in this country, and certainly represents an evolution in simplicity and cheapness of the forced-draft system of ventilation as applied to the drying of soap.

Installation of Disc Fan.—In installing the disc fan where it is set in the side wall, window, or roof, a strong framework of wood mortised and tenoned together is recommended. Secure this framework in the opening in the wall, window, or roof with lag-screws or bolts with screw-nuts; plank over the entire surface of the framework and opening; then cut a circle from the planking the diameter of the flange on the inside edge of the metallic frame of the fan; screw the

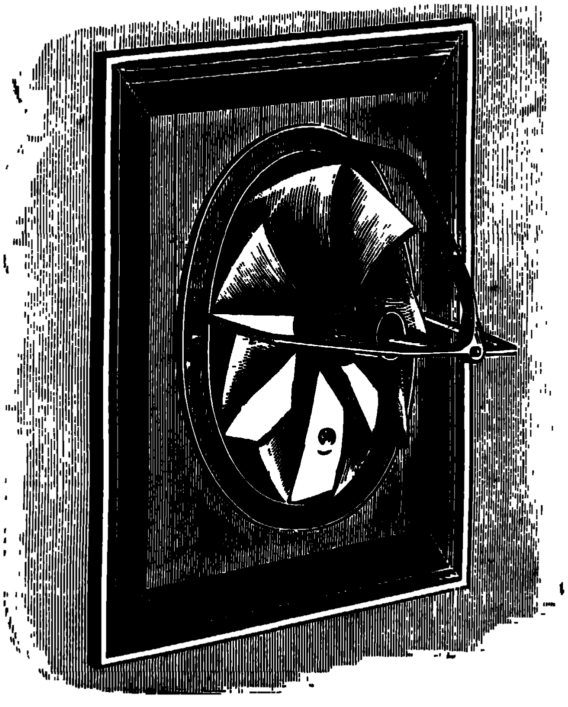


FIG. 97.—Disc Fan as Installed.

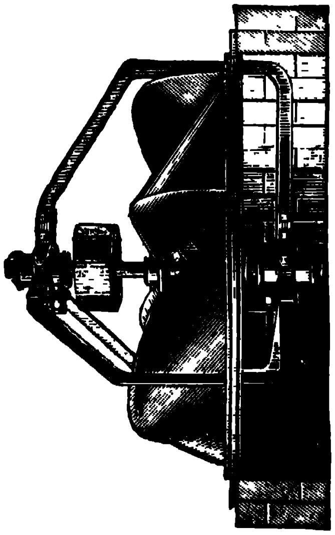


FIG. 98.—Disc Fan Set in the Side Wall.

frame securely to the planking, taking special care that the entire work is set perfectly plumb and true, otherwise it will be impossible to run a belt upon the pulley or to get satisfactory results. When the fan is thus set upright in the side wall or window and ready for the

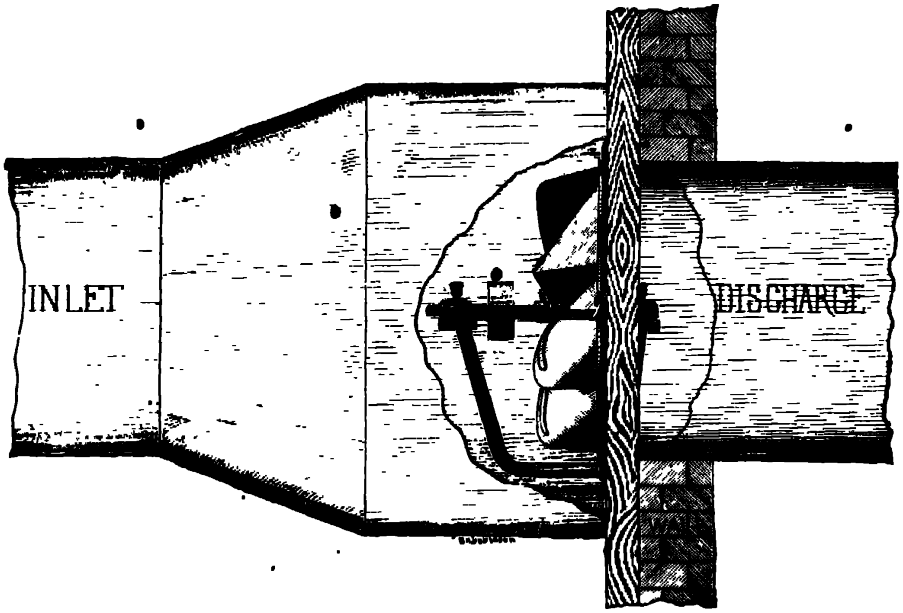


FIG. 99.—Disc Fan Set in the Side Wall, and Interposed in the Air-conduit.

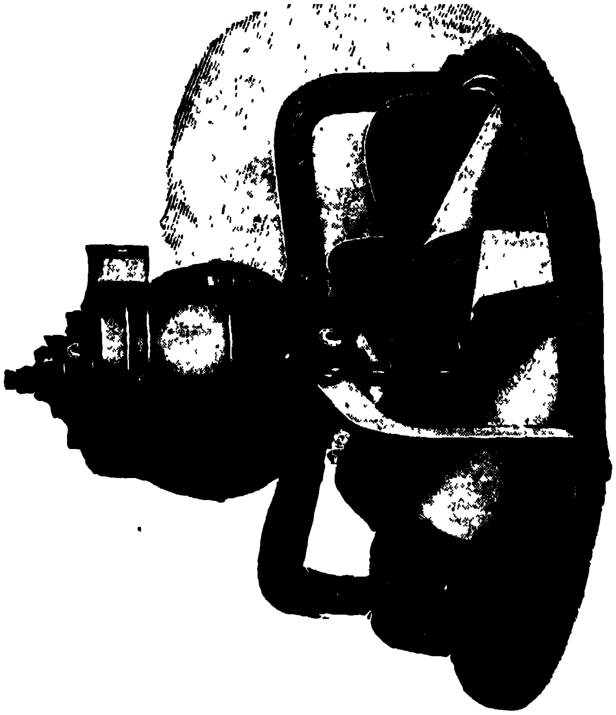


FIG. 100.—Disc Fan Driven by a Motor.

belt to be applied it will appear as shown in Fig. 97. When the fan is set upright in the side wall or window, Fig. 98, and it is desired to pipe back from the fan on the inside and also discharge through a pipe as shown in Fig. 99, care must in every instance be taken that the inlet or supply-pipe be enlarged one fourth of the fan's diameter for a distance back from the fan of not less than the diameter of the fan where it can be tapered down to the diameter of the fan as shown in Fig. 99. When used for moving air by exhaustion care should be taken to see that the fan is the only possible outlet for air. All openings near the fan must be closed when set as shown in Fig. 97. Should openings be left in the drying-room the fan will take its supply from the nearest source and the moist air below the level of the fan will not be drawn towards it. To secure the best results in the drying of soap the openings for the ingress of dry air should be so located as to insure its uniform distribution throughout the mass of racked soap as it is drawn to the fan. In Fig. 100 is shown a type of disc fan driven by a motor.

The Foot-press.—The foot-press, shown in Fig. 103, with mechanism for elevating the lower die on removal of the pressure and with safety-guards to prevent injury to the fingers of the operator, is in its rudiments the simple hand-stamp, Fig. 101, applied to the dried cake of soap contained in a metallic box, to which shape the cake is made to conform by pressure applied by the foot of the operator. The foot-press provides a sudden powerful blow upon the cake of dried soap placed in the box of the die with the thumb and index or second finger of the right hand, the left hand being used in the same manner to remove the pressed cake as it is forced out of the box by the upward movement of the lower die. Power is applied by the foot of the operator and is aided by a system of counterpoised weights which after the blow has been delivered return to their original position, at the

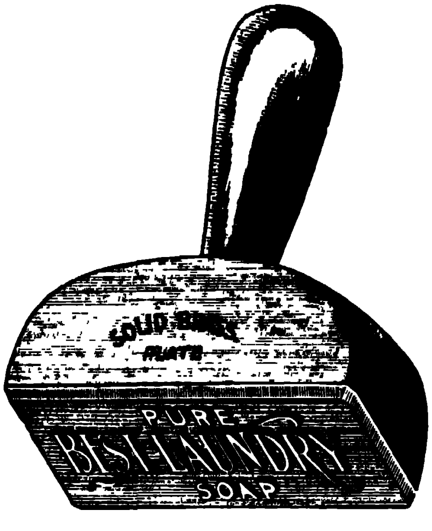


FIG. 101.—Hand-stamp.

same time expelling the pressed cake of soap from the box. By reference to Figs. 102, 103, and 104 it is seen that a foot-press consists essentially of a bed-plate substantially supported and suitably grooved

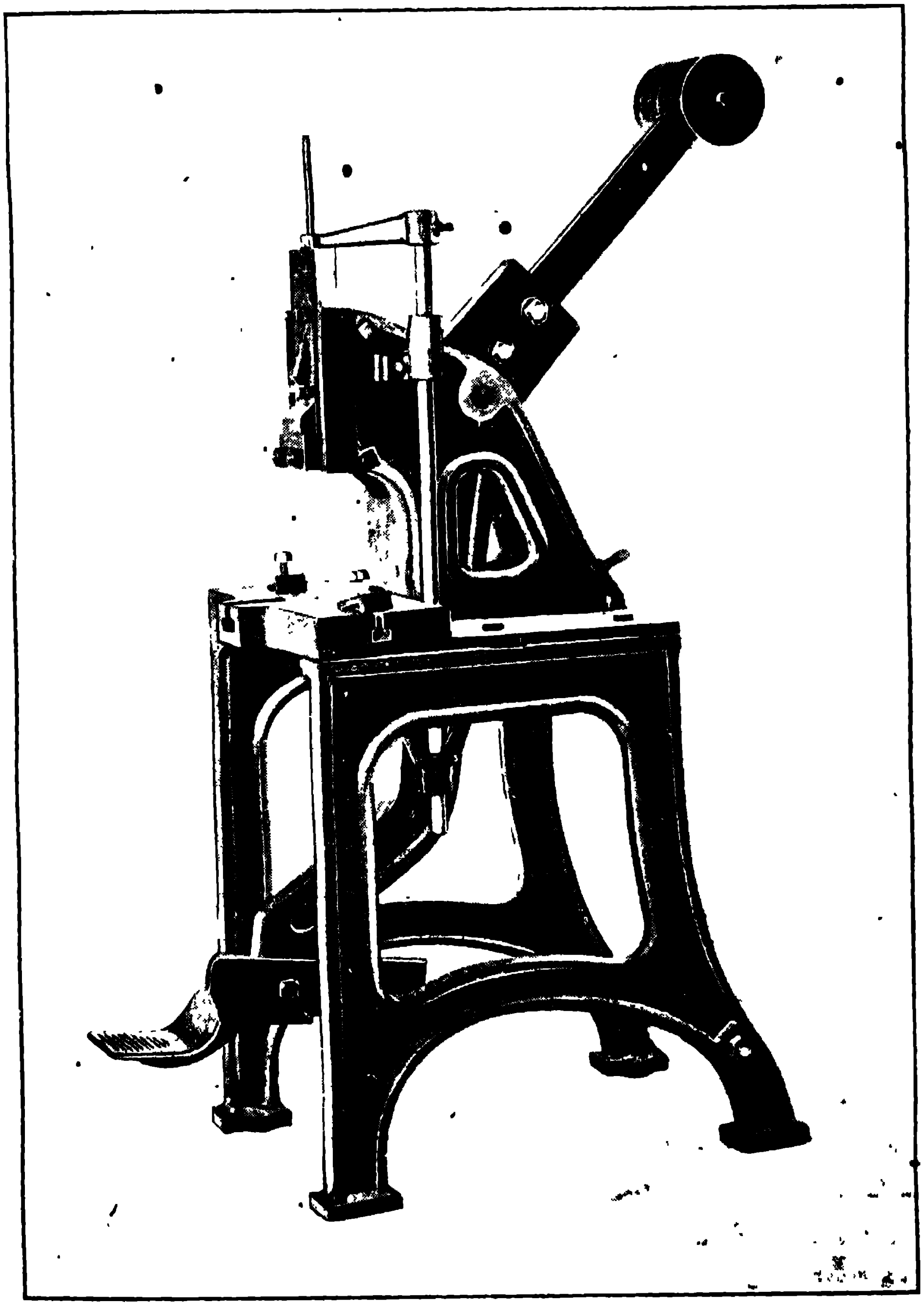


FIG. 102.—Foot-power Soap-press.

for the firm clamping of the die-box, a movable plunger for the elevation and depression of the lower die, and a vertical shaft moving truly in a guide and to which is attached the shank of the upper die.

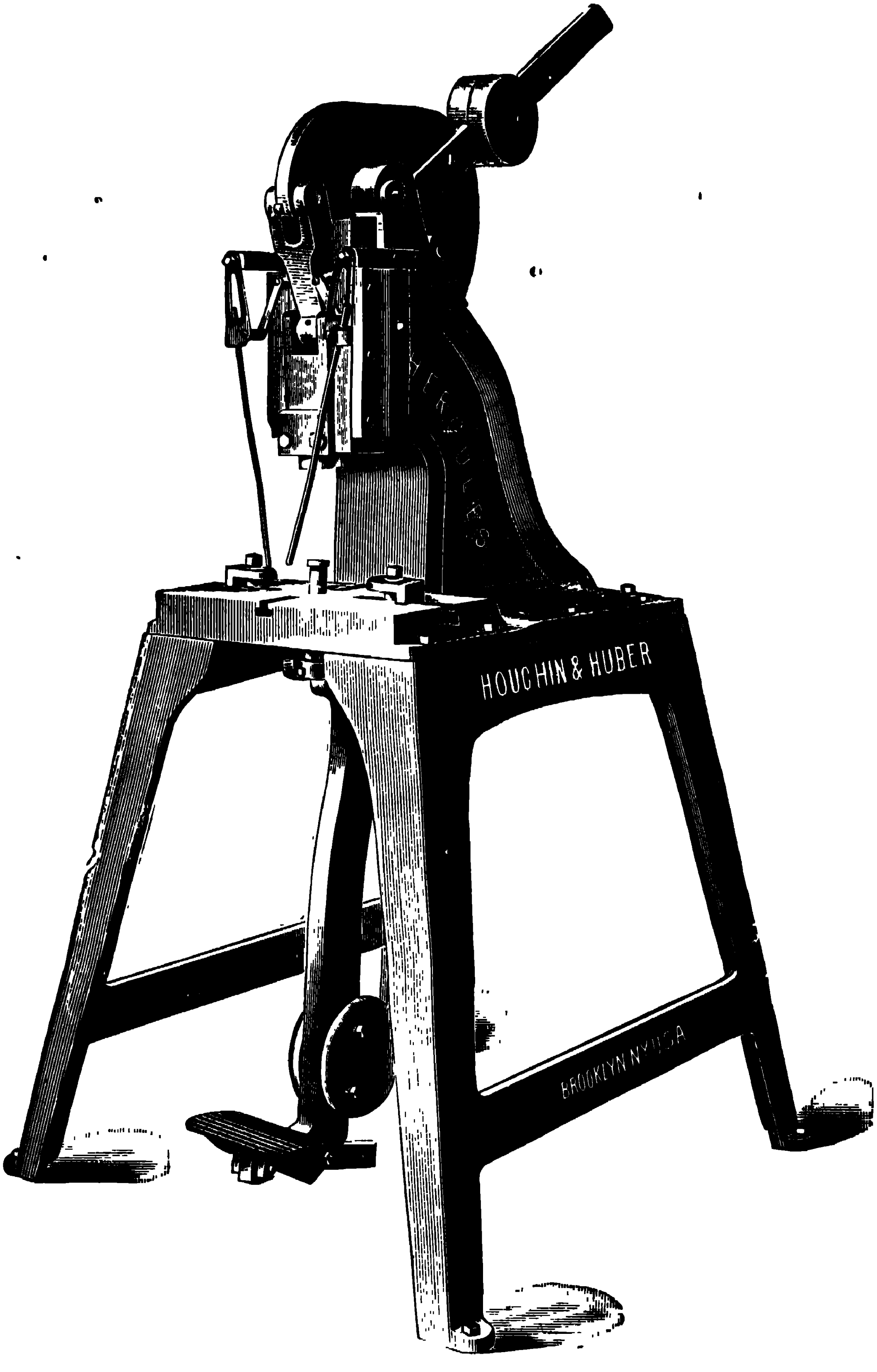
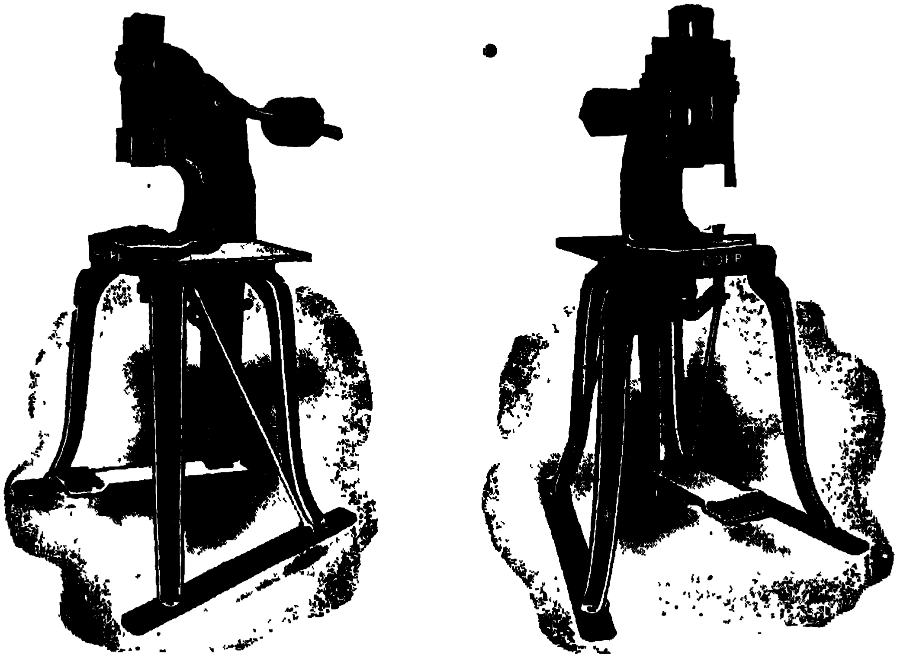


FIG. 103.—Foot-power Soap-press.

By suitably adjusting the counterpoised weights the power of the blow can be adapted to soap of any texture or degree of firmness.

By attaching a single-action steam-cylinder below the bed-plate in such a position that its piston by means of a cam actuates the lever bearing the counterpoised weight, steam may be successfully applied to the operation of a foot-press.

In the type of foot-press shown in Fig. 104 the ram is provided with four V-shaped planed slides, which run in corresponding babbitted ways; two on the head proper, the remaining two on an adjustable cap-plate. By means of this plate, absolute adjustment is always obtained, which means perfect alignment, and long life to the die.



No. 1. No. 2.

FIG. 104.—Foot-press.

The knock-out pin, whereby the pressed cake is ejected from the die-box on the upward movement of the ram (upper die), is operated by a cam-action. Press No. 2, Fig. 104, is provided, as shown, with three knock-out pins, as well as guide- or steady-rods on either side of the ram for the purpose of pressing long bars.

The following considerations are important in determining the choice and use of a soap-press: requirements of the factory as to the amount and variety of the output; ready adaptation of the press to dies of different types; ease of operation; noiselessness; stability; the guide for dies must be perfect to eliminate undue wear, and the arrangement for lifting the cake from the box must be such as to prevent the defacing of the impression by too forcibly ejecting the cake against the upper die.

Pressing Soap.—To press satisfactorily, the soap must be in proper condition. This is a matter of great importance in the use of automatic power-presses, wherein from their nature there can be no variation in the power of the stroke; while with foot-presses the operator may adapt at will the intensity of the stroke or blow to the character of the soap.

As will be learned in the discussion of the boiling of settled grained soap, the settling change is the crucial stage of the entire procedure of soap-manufacture. In considering the settling change at this time from the point of view of soap-pressing, we may state in general that the settling change is the resultant of all the influences that have affected the soap during the prior procedure. If the soap is not well made it cannot be settled satisfactorily. If the soap is not settled properly, taking into consideration the character of the stock and the consistency desired in the finished product, the defects are bound to show themselves in each successive stage, and finally in pressing. Confining our attention entirely to the behavior of soap in pressing, we may state that a firm-bodied soap should be settled "thinner" or "finer" than a softer-bodied soap. Soap from firm-bodied stock, settled coarsely, dries quickly on the racks, lacks flexibility, and, under the sudden, powerful blow of the press, will invariably fracture, with a resulting large proportion of scrap, to be reworked, abuse of the soap-dies, added burden upon the operator, and lessened output.

Soap from soft-bodied stock, settled thinly, to say nothing of the large nigre which invariably will form, if it form at all, dries slowly on the racks, is soft, and adheres to the soap-dies. While there may be no loss in scrap such as characterizes the other extreme before described, equally unsatisfactory results in pressing will obtain.

These remarks are made to indicate the influence of an improper settle on the behavior of soap in pressing.

In the manipulation of milled toilet-soap prior to pressing, as will be described in the manufacture of soap of this class, the consistency of the soap with respect to pressing must be constantly born in mind.

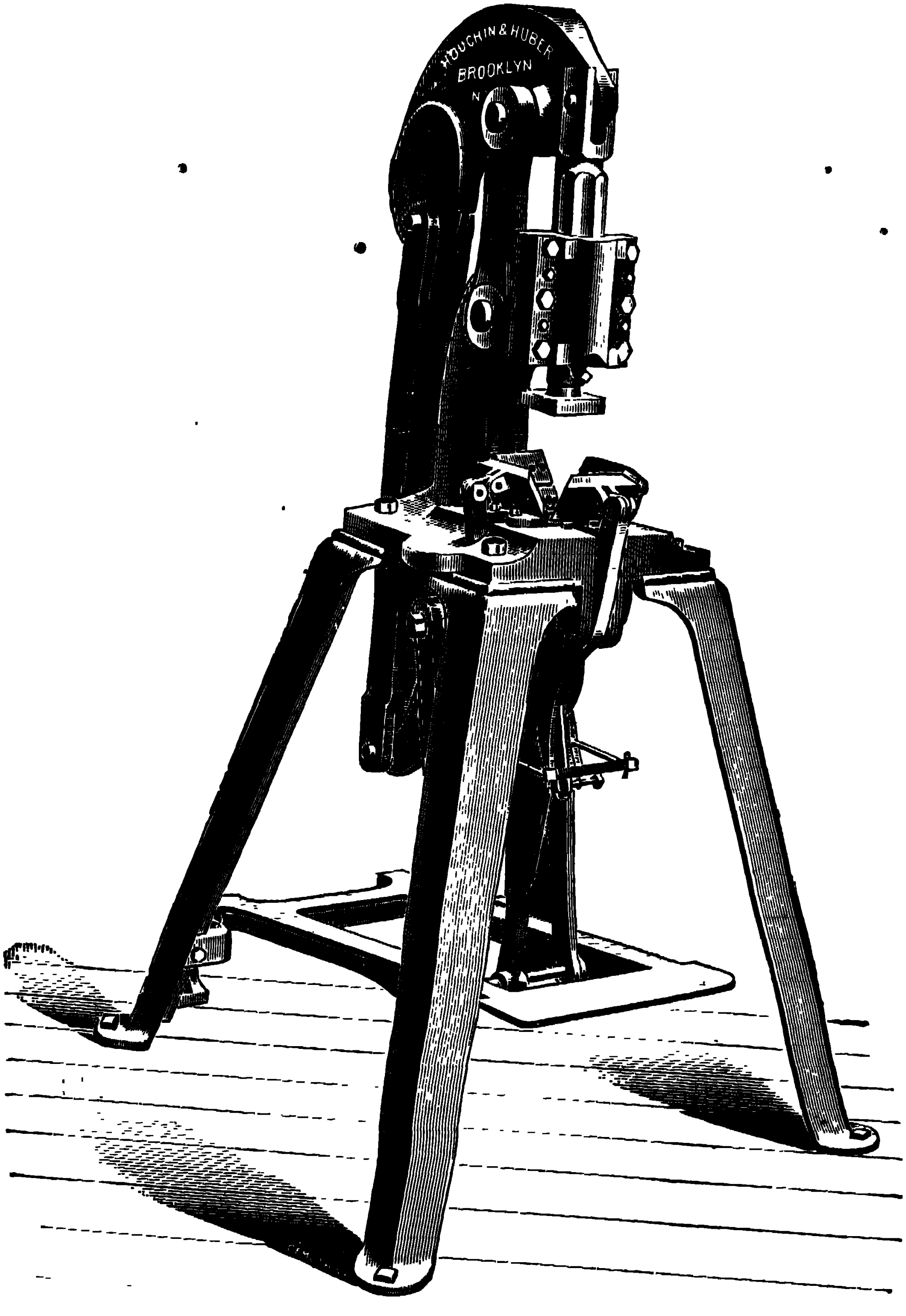


FIG. 105.—Foot-power Press for Impressions on all Sides of Cake.

Cold-process soap, from its nature, is very satisfactory to press.

Scouring or sand soap gives comparatively little trouble in pressing. When slabbed and cut for pressing in a foot-press it should not be allowed to become too dry. This observation, while important, is of lesser consideration when the scouring-soap is moulded and the multiple-die press is used.

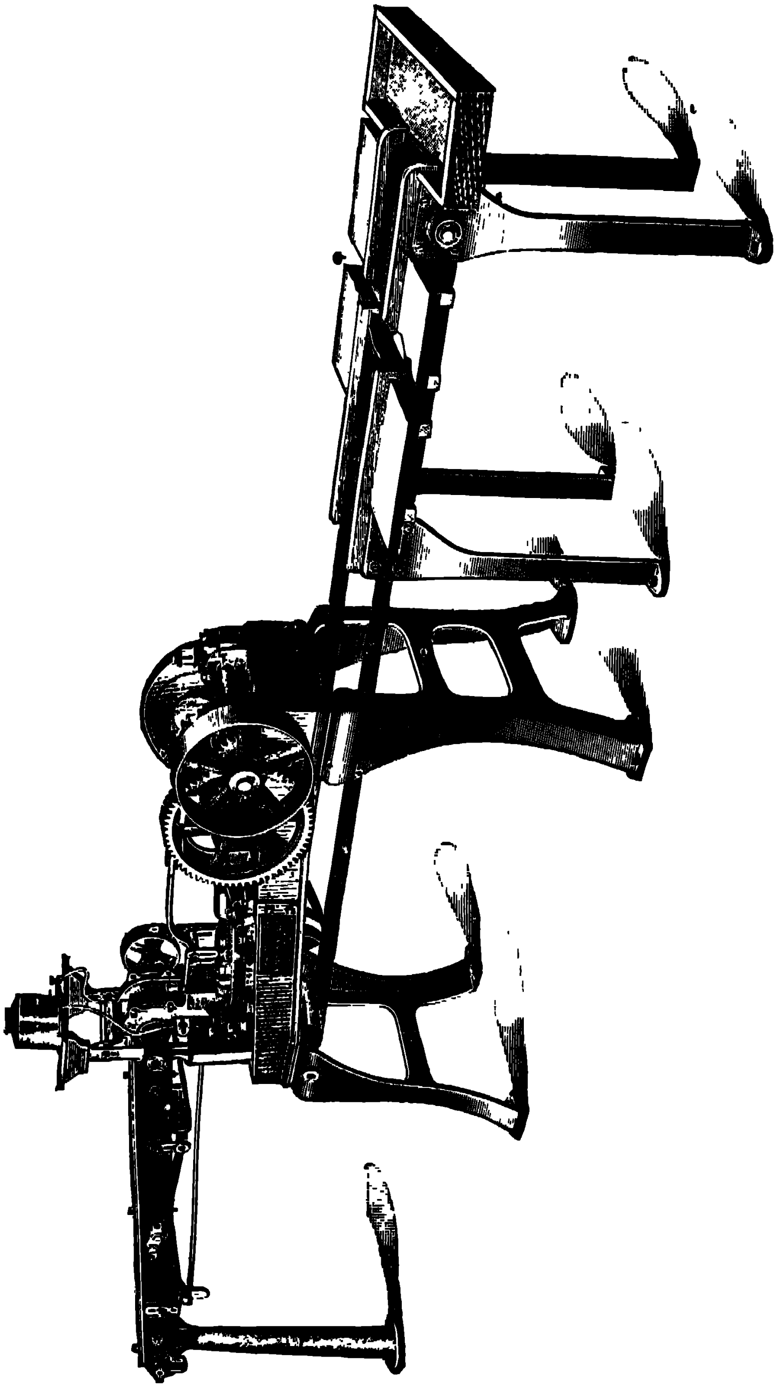


Fig. 106.—Duquesne Interchangeable-die Soap-press.

The lubrication of the dies is more or less essential with all soaps, according to their consistency, to prevent the soap from adhering to the

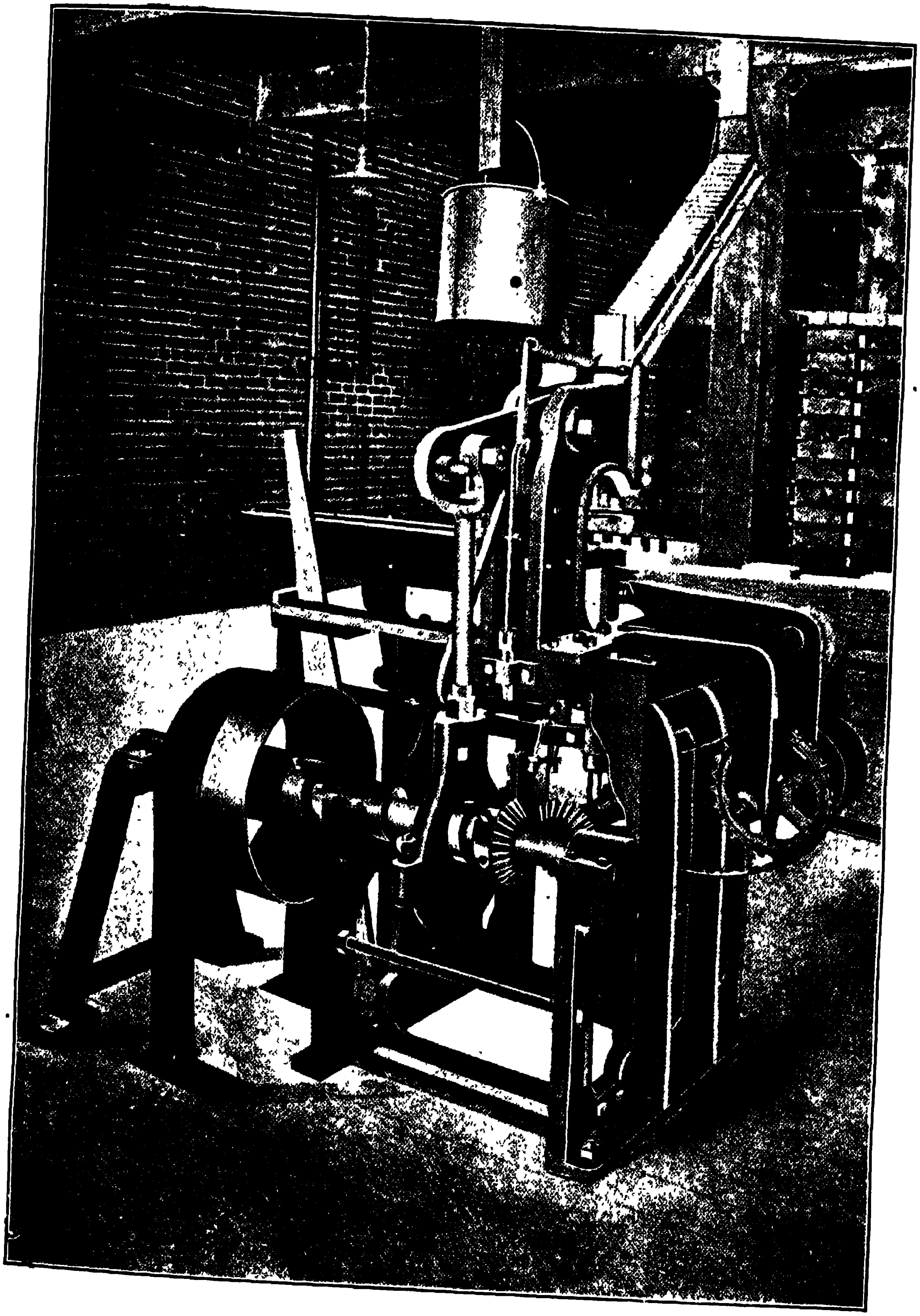


FIG. 107.—Detroit Automatic Soap-press.

lettering or other design of the dies. It cannot be dispensed with in the use of the automatic power-press wherein the lubricating liquid is allowed to drop by gravity upon the soap and dies from a suitably ocated reservoir; with the foot-press the necessity of cleaning the dies, usually with a brush and water or brine, is determined by the consistency of the soap.

A foot-press for the pressing of six-sided cakes is shown in Fig. 105.

The output of a foot-press depends upon the shape and size of the cake, the condition of the soap, and the skill of the operator. If the soap is too moist it adheres to the dies; if too dry, it tends to crack, which is especially true of all short-texture soap, such as filled mottled soap. In any case, more or less cleaning of the dies with water, pickle, or dilute acetic acid is necessary.

One hundred boxes a day, one hundred cakes to the box, if pressed good and clean, is considered a fair minimum day's work. Some factories do not look for their pressmen to work so rapidly and prefer their men to do less, but to turn out well pressed soap.

Soap-pressing by manual labor is subject to many disadvantages, chief of which is the common liability of the operator to loss of fingers through carelessness.

The Power-press.—At present the automatic power-press represents the last stage in the mechanical evolution of the hand-stamp. It has arisen to supply the demand for a press that would be safe, rapid, and economical. The duplication of the old-time foot-presses and the excessive amount of labor for their operation have been sources of expense and annoyance in many ways. In Figs. 106 to 112 are represented five forms of this type of press. For their satisfactory operation the soap must be well dried and lubricated. Early forms of the automatic power press were not adapted to the use of different dies, but the more successful forms at present in use possess the interchangeable-die feature. In the Duquesne interchangeable-die press, Fig. 106, the cakes are transferred by hand from the racks on to an endless belt, running on the surface of the feeding-table, whereby the cakes are automatically fed into the drop-box, down which it falls between two dies impelled in a horizontal plane. By the reverse operation of the upper and lower dies, the pressed cake is withdrawn from the box and then by the operation of suitable grippers the cake is so clamped or held as to permit of the separation of the movable dies, acting as plungers working horizontally from the sides of the cake of soap. The pressed cakes drop upon an endless belt and are carried to the wrapping-bench.

In the Detroit press, a view of which is shown in Fig. 107, power is transmitted to the upper and lower dies, moving in a vertical plane, by means of an eccentric attached to the shaft bearing the belt. The cakes are fed to the die-box by gravity, being placed in a feed-box supported at an angle of 45 degrees. The cake is thrust into the box from beneath the pile of superincumbent cakes by means of an automatic pusher, which, together with the unpressed cake, throws the pressed cake as it is elevated by the lower die on to an endless belt, by which it is carried to the wrapping-bench.

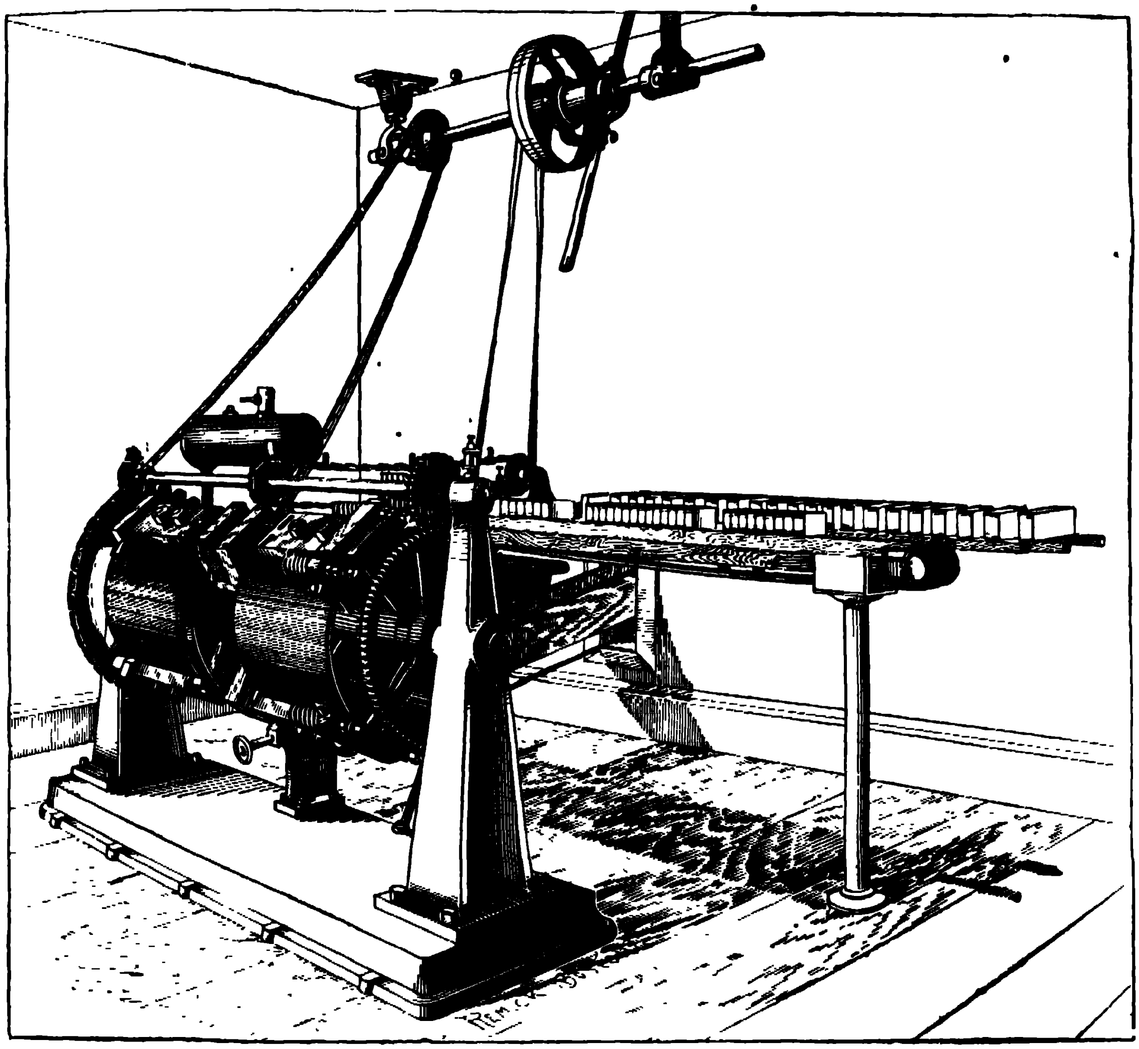


FIG. 108.—The Curtis Davis Three-die Rotary Press.

One of the earliest successful automatic soap-presses is the

Curtis Davis three-die rotary press shown in Fig. 108. It consists essentially of double cylinders placed end to end, in which rotate cams that automatically bring together and withdraw horizontally two dies, corresponding to the upper and lower dies of the foot-press, which meet in their corresponding die-box, of which three with their corresponding dies are arranged 120° apart. The dried soap is transferred from the racks on to the feed-belt, from which at right angles each bar is singly moved forward to the die by means of a "finger" on the small chain-belt shown in the figure and to which power is applied by the large chain-belt suspended from the shafting. The pressed cake is dropped on a belt which carries it to the wrapping bench.

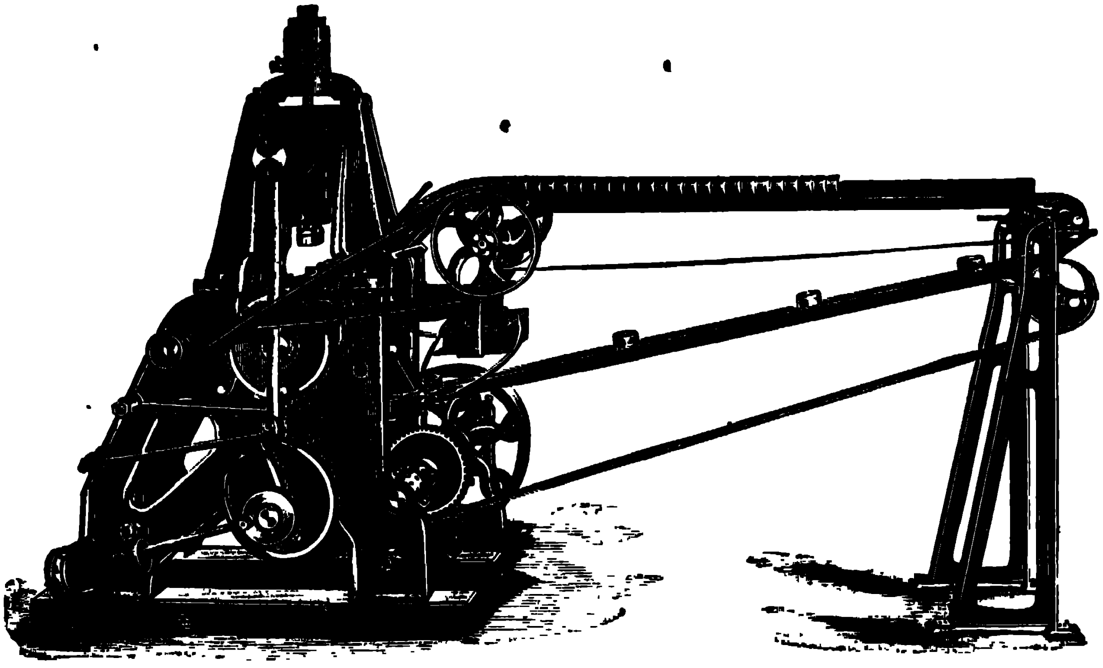


FIG. 109.—The Tilden Automatic Power-press.

In the Tilden press, Fig. 109, the cake, placed on its narrow side on the feed-belt, is projected into the die-box, where it is subjected to the pressure of vertically moving dies. On the release of the pressure the pressed cake is automatically elevated by an upward movement of the lower die, whereupon it is automatically displaced and dropped upon an endless belt.

The Buffalo automatic power-press, Fig. 110, is actuated by means of suitable gears and cams driven by belt. The cakes of soap are fed into the inclined guide. The lowest cake in the guide is pushed automatically under the upper die, which movement is properly adjusted with that of the upper die. After the first cake is passed under the die, the second cake is pushed forward and ejects the first cake, which has been pressed and raised out of the die-box. The average rate of operation is 165 cakes per minute. The standard foot-press die, with 1-inch shank, is available for use without change.

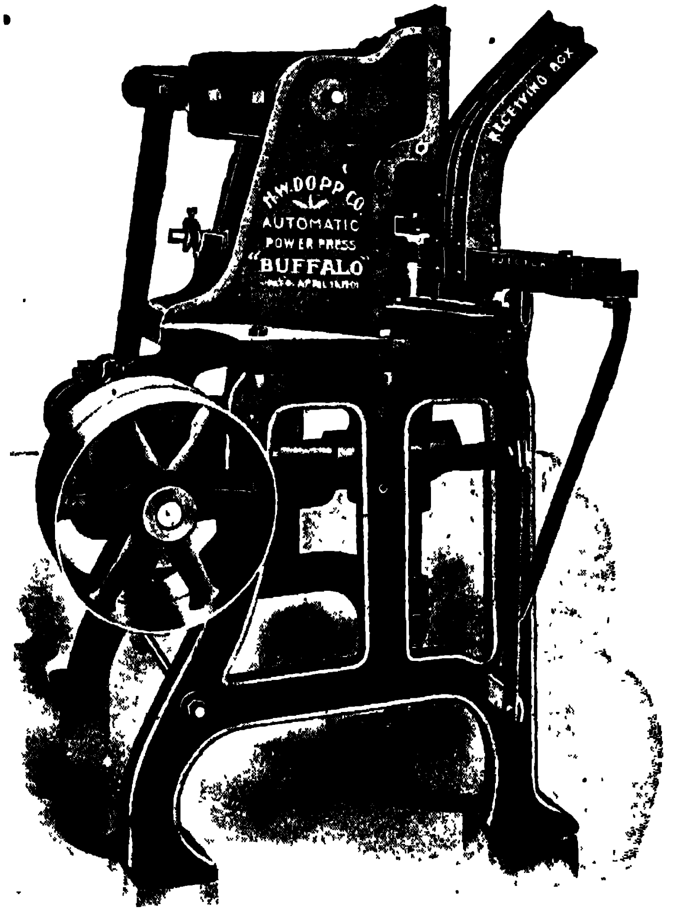


FIG. 110.—The Buffalo Automatic Power-press.

As volume of business determines the use of automatic devices of this character, they are found only in the largest manufactories or where a large amount of a single brand is pressed. They vary in capacity from 500 to 800 boxes per day, representing an output of 5 to 8 foot-presses and requiring the labor of but one attendant.

The Miller press, Fig. 111, is representative of a class of power-presses which, while preserving the mechanical features of the foot-power press, effect the application of pressure either by steam directly or by belt. In the Miller press, power is derived wholly from the belt, the operator being required merely to place the cake

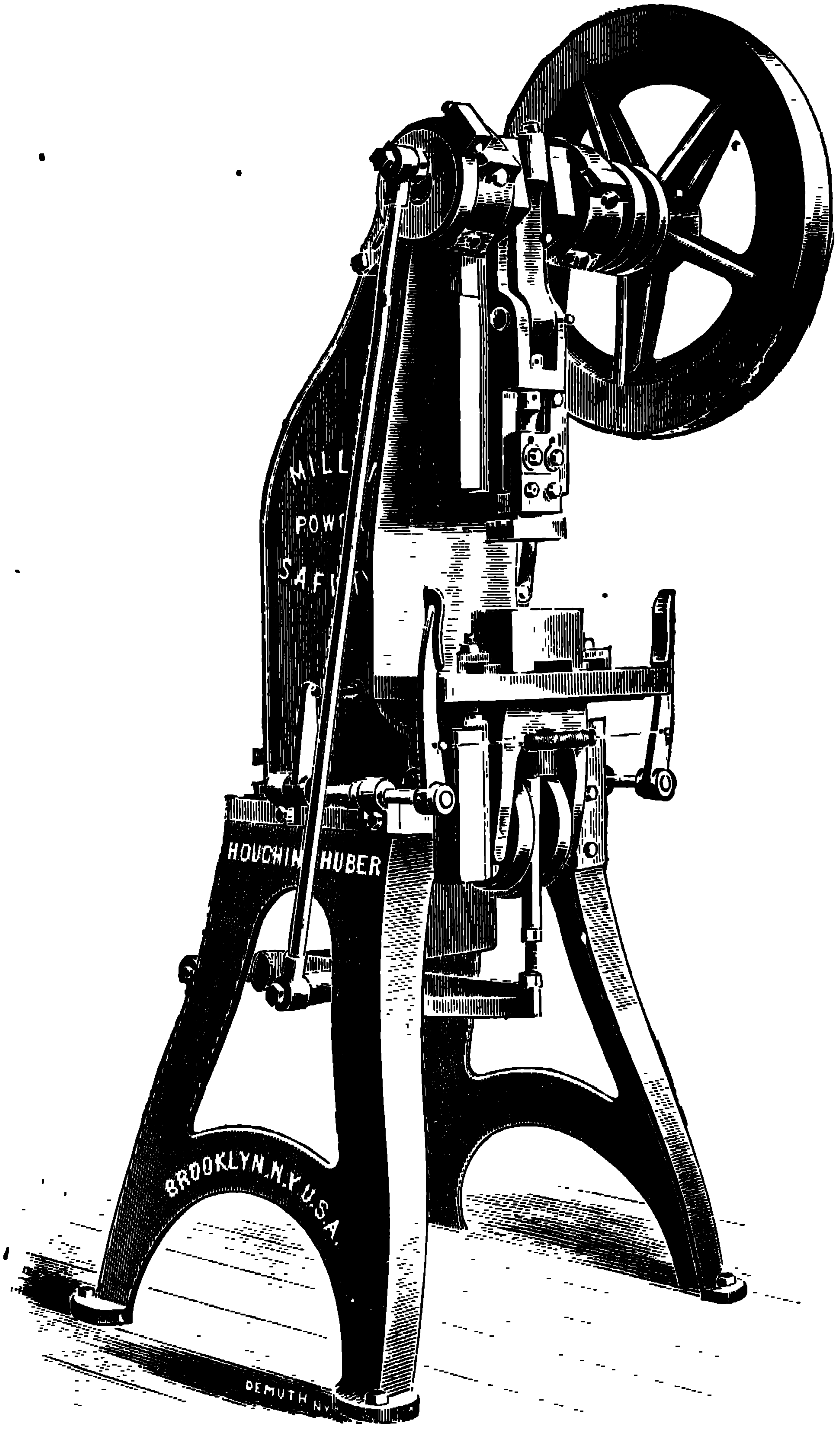


Fig. 111.—The Miller Safety Automatic Power-press

in the die-box, as is customary with foot-presses, and to remove it in the same manner.

Its safety to the operator lies in the fact that the blow that gives the impression cannot be made without using both hands to push the levers, shown on either side of the bed, outwards. This double movement, after a little practice, is simultaneous with reaching for and depositing the cakes of soap.

As the impression is made one-fifth of a second after the levers are touched, the blow is practically instantaneous, not giving the operator time to bring his hands back again to the dies.

Any set of dies that are now used in foot-presses can be used in this press, compensating devices for thick or thin cakes being provided.

The press is also arranged that cakes of varying thickness will receive the same pressure, as the bed of the press is movable, being controlled by a weight not seen, and which leaves the force of impact regulatable by the adjustment of this weight.

The operator may be provided with a bench or chair to sit upon while pressing, and a helper to carry the soap to the presser may be employed to keep the machine at its maximum speed. In this manner 100 to 125 gross of toilet-soap, for which the press is especially adapted, may be pressed per day, without any injurious or arduous effect upon the operator.

Soap-dies.—Previous to the introduction by Babbitt of the bar soap, such as we are familiar with to-day, soap was sold in bulk by weight, just as the grocer now dispenses cheese. An improvement upon this awkward manner was cutting the soap into pieces of convenient size and marked with the name of the manufacturer. These pieces were either wrapped or unwrapped. This style, plainly marked with the hand-stamp, persists to-day in many localities. This improvement in the form of soap placed upon the market paved the way to the introduction of the cushion-shaped pressed bar, and with this innovation we may date the beginning of the soap industry as we know it to-day.

There are four classes of soap dies, viz., the hand-stamp, the progenitor of all others, shown in Fig. 101, the box-die, Fig. 112, the pin-and-shoulder die, Fig. 113, and a combination of the box and pin-and-shoulder dies, Figs. 114 and 115.

The hand-stamp is the earliest form and is used for stamping unmoulded soap with the brand and name of the manufacturer or vendor. It consists simply of an engraved brass plate, mounted on a wooden base, provided with a handle. The three succeeding forms of dies are essentially moulds in which the soap is compressed by the dies simultaneously with the impression. The common form is the box-die, Fig. 112, which is used universally for laundry soap. It consists essentially of a box in which the dried soap is compressed by the upper and lower dies; the stationary box, bolted to the bed-plate of the press through the shanks, and the movable upper and lower dies constituting the mould. By varying the height of the box or the thickness of the lower die, which, however, should be as high as permissible to provide a large bearing surface, cakes of different thickness and weight can be pressed in the same box. By means of movable die-plates or panels in the lower die, Fig. 115, different impressions can be made in bars of the same size. The movable dies are machine fitted to move accurately in the box, but commonly, owing to imperfect adjustment of the dies and faulty movements of the press, which arise with long-continued use without skilful repairing, the accuracy of the dies is often soon destroyed and the upper one rendered worthless for good work. To overcome this inequality of wear, a self-adjusting die is made in which the box is provided with projecting lugs or shoulders drilled to receive the two gauge- or guide-pins which project downward from a corresponding shoulder on the upper die.

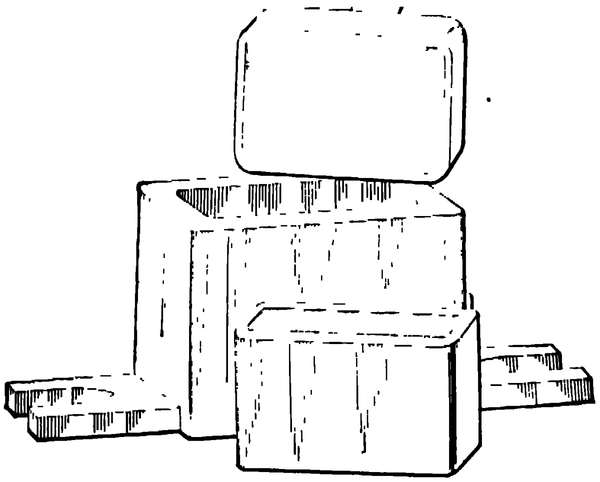


FIG. 112.—The Box-die.

As these steel pins project beyond the lowest point of the upper die and enter the steel-lined recesses in the shoulder of the mould before the upper die reaches it, they compel the uniform entrance of the upper die into the mould and obviate any possible chance of nicking or shaving. This improvement, together with a section through an ordinary box die, is shown in Fig. 115.

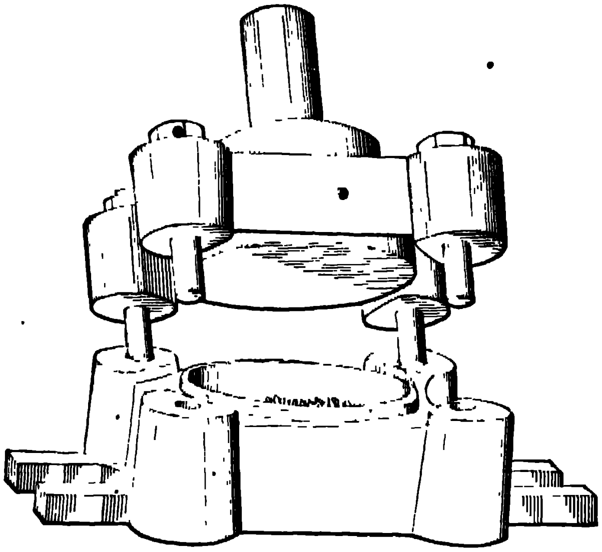


FIG. 113.—The Pin-and-shoulder Die.

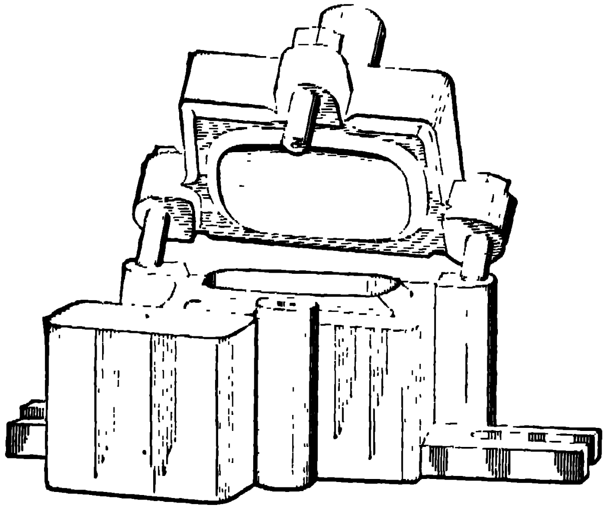


FIG. 114.—Combination Box and Pin-and-shoulder Die.

The pin-and-shoulder die, Fig. 113, is used exclusively for milled soap. It consists of two parts, viz., the upper, movable die carrying projecting lugs or shoulders with guide-pins, and a lower stationary part, forming the mould, carrying shanks for attaching it firmly to the bedplate of the press and shoulders with corresponding holes for the reception of the guide-pins of the upper die. The shoulders receive the force of the blow and allow the dies to meet accurately at the cutting-edge, whereby all superfluous soap is removed. In the box-die all the soap in the cake is compressed into a bar, the pressure of the blow conforming the cake to the mould. With dies of this class all cakes are pressed to a uniform weight, all surplus soap being separated by the cutting-edge.

The combination die, Figs. 114 and 115, is simply an adaptation of the box-die, whereby its use with milled soap is possible. Not only is all surplus soap expelled, but by means of a lower die of variable thickness, cakes of different weights can be pressed. Regarding the dies, it is of importance that they should be constructed and arranged with a proper regard for the particular purpose for which they are intended; in other words, they must be adapted to the grade of soap for which they are to be used. The lettering of the die must be made so that it will not retain a hold on the soap, but should release or withdraw from the soap easily, and the fine, sharp points of a design should be made as large as the circumstances will permit, in order to give such places the greatest strength possible. The workmanship of the die is shown at once in the appearance of the pressed soap. The engraving should be skilfully executed, so as to produce in the soap clear-cut and even letters, especially when the characters on the soap are in relief. All surfaces in contact with the soap should be perfectly smooth in order to produce, especially with milled soap, a smooth and glossy surface. With a die of inferior workmanship, a good appearance cannot be imparted to any soap, whatever its quality; on the other hand, a finely executed die greatly adds to the appearance of an inferior product. Soap-dies are made usually of gun-metal or rolled brass. The box may be made of iron and lined with brass.

In Fig. 116 is shown a die-box of novel construction to permit the taking up of inevitable wear and to insure accurate adjustment of the movable upper and lower dies to the stationary box. As wear ensues with a box of this design, it will not become necessary to spread the edges of the dies to make them fit the box, but plates may be removed to effect the adjustment necessary.

Soap-wrapping.—The pressed soap is delivered from the auto-

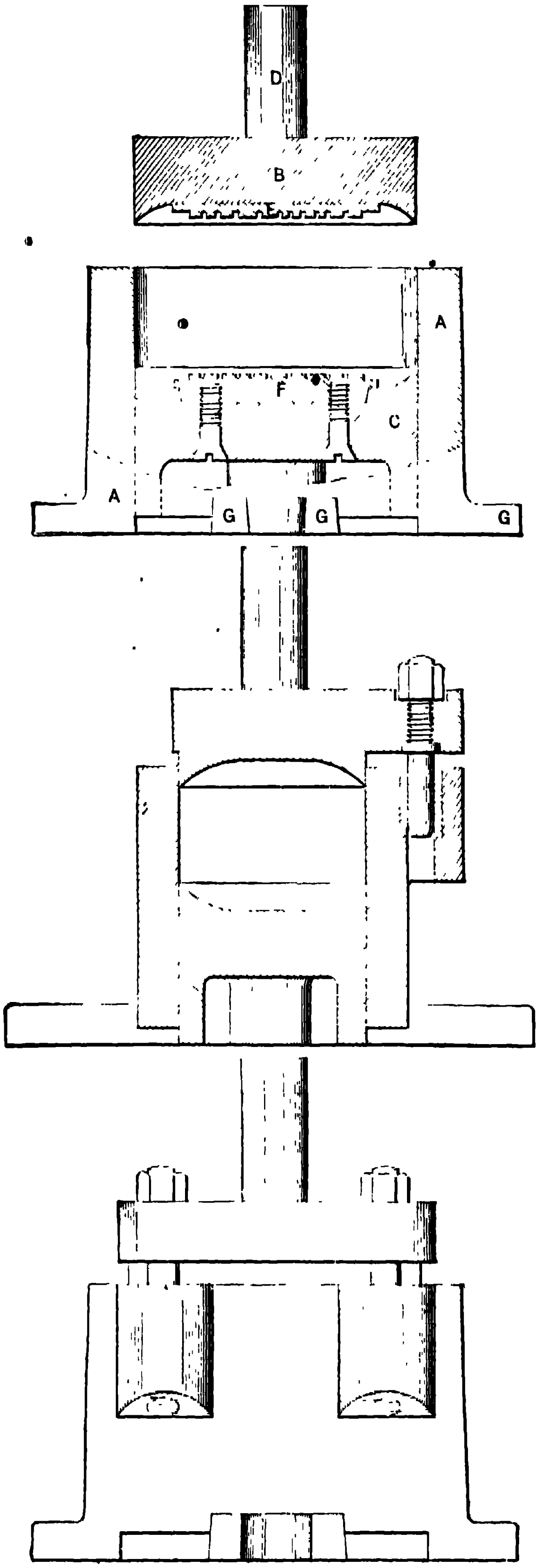


FIG. 115.—Combination Die. (A, box, *B*, upper die; *C*, lower die; *D*, shank *E*, fixed panel; *F*, changeable panel; *G*, flanges.)

matic press by belt conveyer to wrappers at a long table, arranged on both sides of the belt, from which soap is taken as desired. Specialization of labor at this stage takes a variety of forms in different plants. Where one person wraps, packs, and nails each box, 40 boxes per day per worker is an average output. In Fig. 117 is shown a type of wrapping-table adapted for use with an automatic press. Soap-wrapping is now effected by automatic machinery, one form of which is shown in Fig. 118. It is automatic throughout, of interchangeable parts, and will wrap round, oval, or square-cornered cakes with both inside and outside wrappers to the extent of 300 to 450 cases of 100 cakes per day. It receives soap direct from the press and conveys it either wrapped to the packing-bench or automatically packs it in the box.

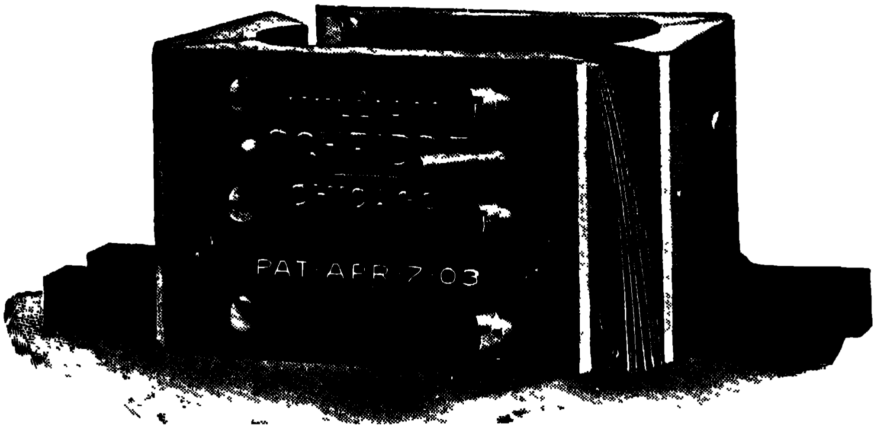


FIG. 116.—Adjustable Soap-die.

The Soap-box.—The question of box supply is an important one with every concern in the industry. With factories located in manufacturing centres, the supply may be obtained from a box-manufacturing establishment. Aside from this advantage the question of procuring box material in shooks in car-load lots may be an important one on the score of economy. With factories remote from manufacturing centres, the purchase of the box in completed form is out of the question, owing to the bulk of the article and high freight. Where boxes are purchased in shook form the question of automatic nailing machinery for the manufacture of the complete box from its parts is to be solved by the volume of production.

Where the volume of business is large, much ingenuity is displayed in the use of labor-saving devices for the handling of empty and filled boxes, as shown in Figs. 118 and 119.

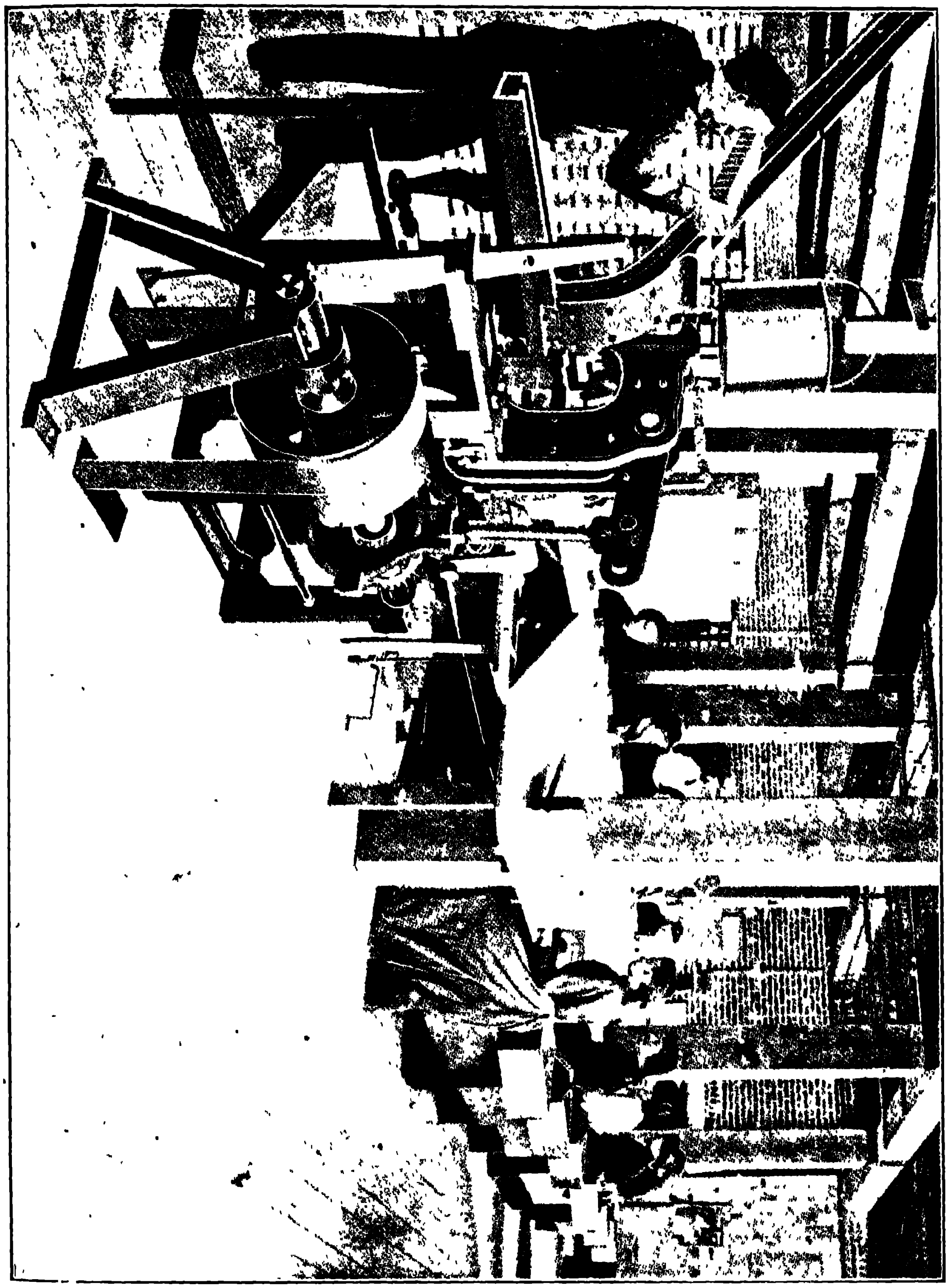


FIG. 117.—View of Automatic Press and Wrapping-table.

Fig. 118 is a diagram showing a method of handling boxes a the Procter & Gamble factory at Ivorydale, Ohio. Empty boxes, *EE*, etc., are brought from the box factory on cars *B* or *B'*, and are delivered by the conveyer *A* (which may be extended to position *A'*) to a slide *Q*, which reaches to the packers. After they are filled and nailed, a conveyer *P* (not shown here) brings them to conveyer *C*, which delivers them to slide *S* in the warehouse. There they are either stacked up or loaded for shipment in cars *R*.

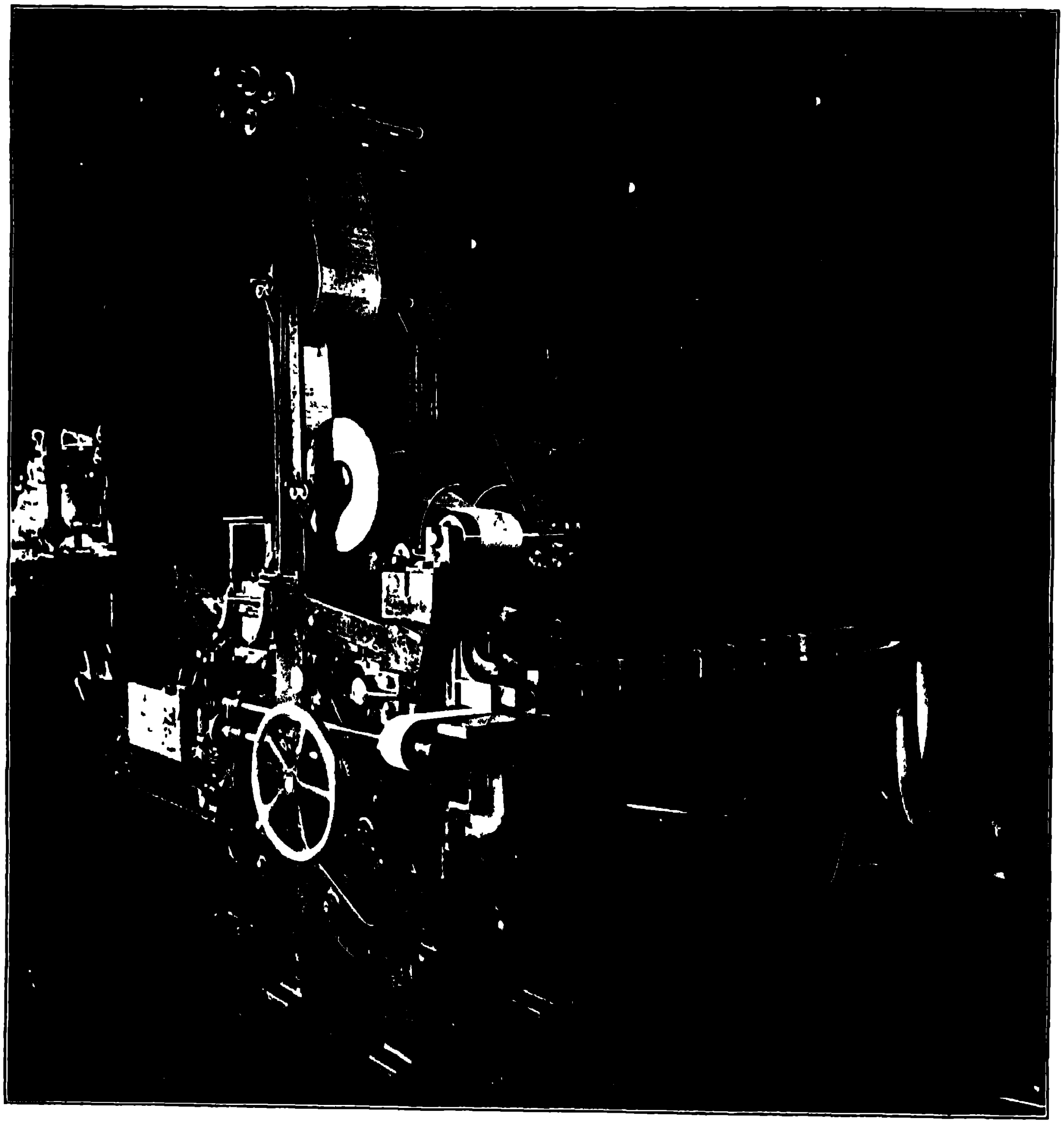


FIG. 118.—Automatic Soap-wrapping Machine.

Summary of Factory Equipment.—We have passed in review the methods of manipulating soap, together with the mechanical apparatus requisite thereto, as they customarily are and have been employed from the inception of the industry. The onflowing tide of inventive ingenuity has modified but little the essential characteristics of soap-manufacture. Should the soap-boiler of thirty years ago, unfamiliar with the mechanical progress that has been made in the interim, return to the scenes of his former laborious activity, he would find no radical change in the essential procedure of manufacture. It is true that where the volume of business warrants, numerous mechanical devices are available whereby labor may be saved and the quality of the product improved. Inventive ingenuity has been directed more to the refinement of long-existing mechanical processes than to the introduction and use of a procedure in any respect comparable to the rapid-tannage processes of modern leather manufacture. Soap in the kettle is a chemical product; soap in the market is a mechanical admixture with the chemical product as the basis. The procedure in the kettle is chemical, after which the procedure is mechanical.

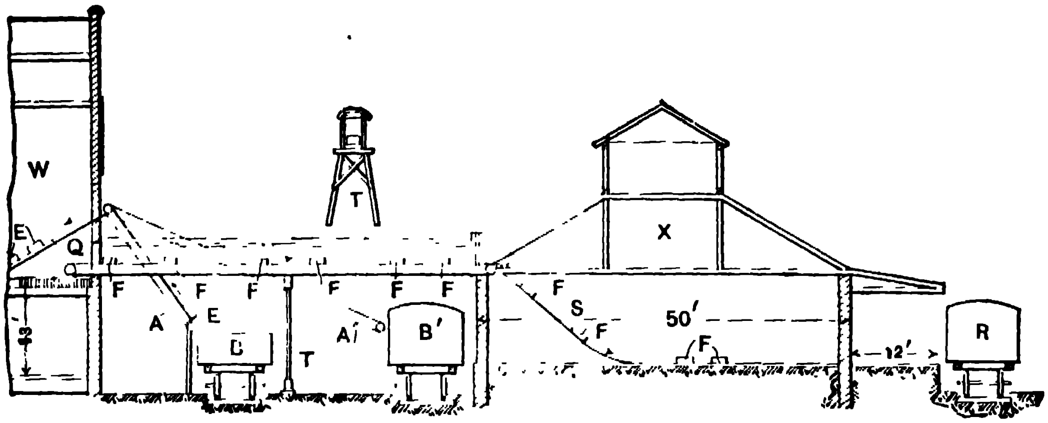


FIG. 118.—Box Conveyers.

Are the chemical and physical qualities of soap such that a period of at least three weeks is necessary to prepare it for sale, or is it that this period is necessary to prepare it as it is desired to make it?

The former question is to be answered in the negative, the latter in the affirmative.

With the exceptions of soap mixed with an abrasive agent (scouring-soap) and soap mixed with a preponderating excess of soda-ash (soap-powder), from a mechanical point of view, soap may be divided into two general classes, viz., laundry soap and milled soap, the former constituting the greatest amount of all soap made.

The qualities desired in the former class of soap is what in popular phraseology may be summed up in the term "a ready washer." This concurrently satisfies on one hand the consumer's demand for ready solubility with a safe degree of neutrality and on the other hand the desire of the soap-manufacturer to increase the bulk and weight of his product with that most inoffensive agent, water. Water with this class of soap represents the soap-manufacturer's legitimate margin.

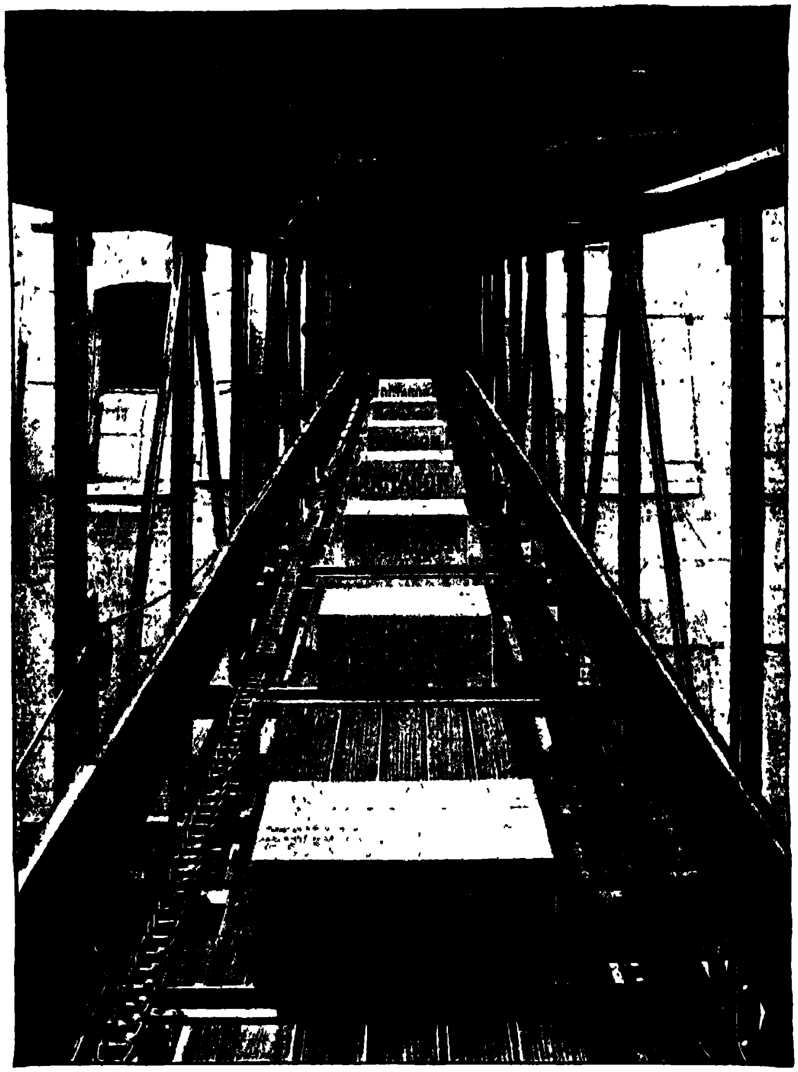


FIG. 119.—Box Conveyer.

Laundry soap as we know it to-day is what it is because it is as the consumer demands it and as the manufacturer happily desires to provide it. Its present form is the resultant of two forces proceeding from opposite directions and in strict obedience to natural law. Things are as they are, not because a particular individual or class of individuals desires them to be that way, but because in their present form they possess their highest present attainable

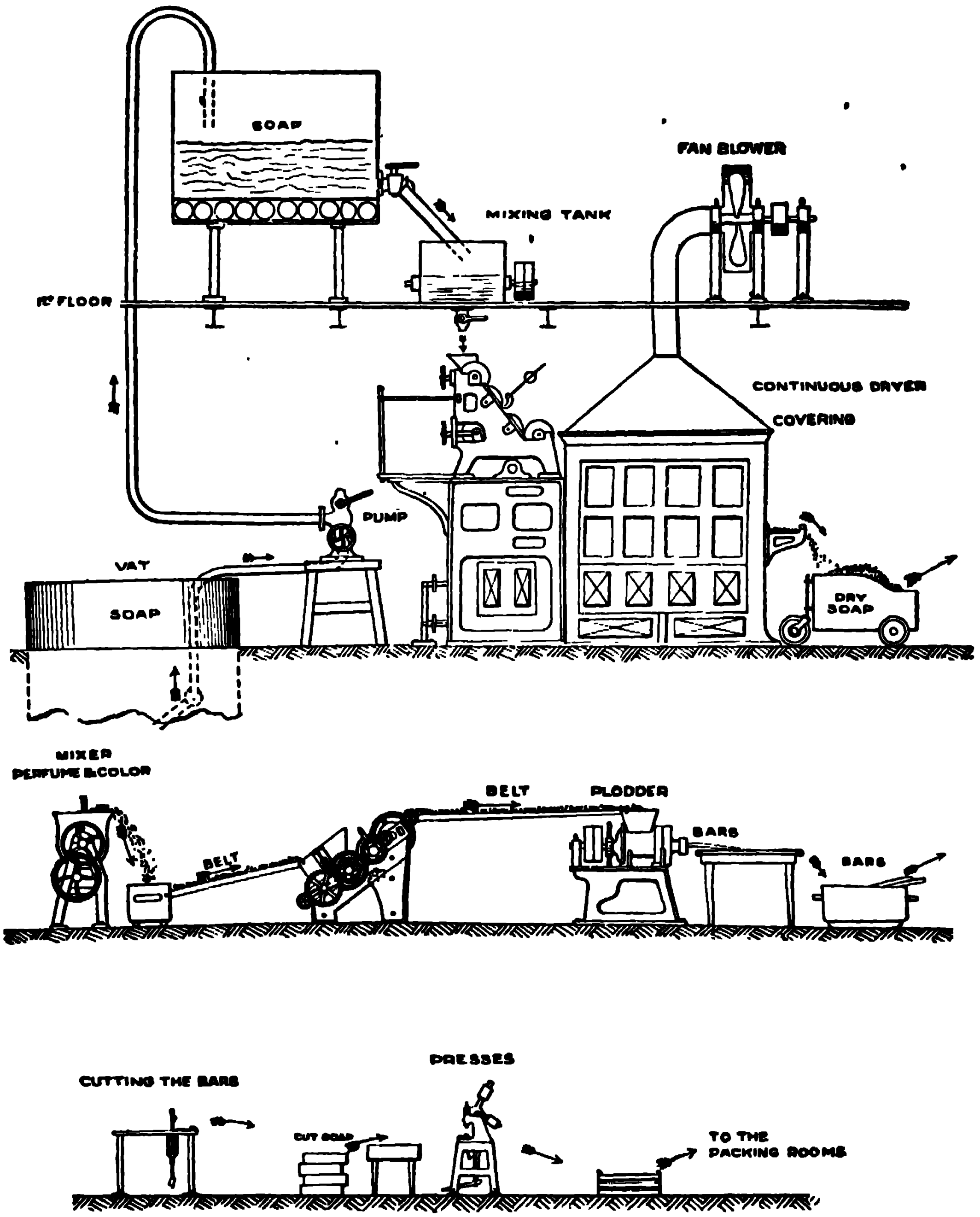


Fig. 120.—Outline of a Procedure for the Elimination of the Present Mechanical Devices Intervening the Crutcher and the Press.

degree of serviceability. This form, however, is by no means stationary. If it were, all improvement would be futile.

If this present form of laundry soap exists solely because soap-manufacturers desire it to produce it in that form, its change would be from motives of self-interest solely a matter of argument *ad hominem.* The present form exists because in that form this commodity possesses its highest degree of serviceability, thereby on one hand satisfying the demand of the consumer and on the other hand the desire of the manufacturer. It is true that the manufacturer would have his margin if the commodity were produced in another form. The material question is, Would that form satisfy the consumer as expressed in the past history of the industry? If that form did not express the highest degree of serviceability at present attainable, it would not carry the seal of popular approval, no matter how ingenious the mechanical device employed to produce it. Only that, whether man or machine, succeeds which serves.

With these ideas in mind, let us return to the consideration of the mechanical procedure following the soap-kettle. It comprises setting-up frames, crutching, framing, stripping, slabbing, cutting, drying, and with milled soap the supplementary processes of chipping, further drying, amalgamating, milling, plodding, and finally the process common to both classes of soap, viz., pressing. Each process exists essentially as it did originally. In each process greater or less refinement of procedure is possible by the use of improved machinery. With laundry soap the introduction of any procedure or device is restricted to that which will not modify or sacrifice the present degree of serviceability of the resulting product. We must conclude that the present field for revolutionary change is very limited. With milled soap, a revolution in the historical procedure has been successfully made, with no change in the character of the product.

If all the forces influencing and determining the character of the product in the former case permit a similar revolution, the economic efficiency of a time-honored industrial process will be greatly increased. In Fig. 120 is shown in outline the essential features of the process as thus modified, and in Fig. 164 the distinctive apparatus of the process.

CHAPTER VII.

COLD-PROCESS AND SEMI-BOILED SOAP.

Cold Process Defined. Purity of Ingredients. Proportion of Stock and Lye. Filling. Temperature. Hand-crutched Cold-process Soap. Apparatus. Mixing. Combination in the Frame. Laundry Chipped Soap. Chipping Soap. Cold-process Soap from Fatty Acids. Cold-process Toilet-soap. Utilization of Scraps from Cold-process Soap. Final Considerations. Modified Cold Process. Toilet-soap: Procedure. Use of Rosin. Tar Soap: Formula; Procedure. Transparent Soap. Ingredients. Processes. Procedure. Semi-boiled Defined. Scouring or Sand Soap. Selection of Stock for Scouring-soap Base. The Abrasive Agent. Killing Stock. Mixing. Frame Method. Mould Method. Scouring-soap Press. Drying Scouring-soap. Soft Soap. Stock. Procedure. Yield.

Cold Process Defined.—The three general classes of soap-manufacturing processes will be considered in the relative order of their economy of operation, intricacy of the manufacturing process, and period of time required for their completion. In this order, the cold process demands first consideration. The term "cold" as applied to this process is a loose descriptive. Saponification is not effected in the cold, and the heat evolved by chemical reaction is considerable. The term refers to the fact that artificial heat is not employed as in the semi-boiled and settled processes, yet to be described.

The cold process consists in the direct combination of the stock with a quantity of alkali, sufficient for the complete saturation of the stock, at a comparatively low temperautre and under conditions that permit of the greatest intimacy of contact of the reacting bodies. As cocoanut-oil and tallow, and to a less degree cottonseed-oil, are the chief stocks employed in this process, their different characteristics require a different set of conditions when used separately and a combination of them when the stocks are used in admixture. Five essentials to satisfactory work with this process are as follows:

1. Purity of ingredients.
2. Accurate proportions of stock and lye.
3. Proper density of lye.
4. Proper temperature of ingredients before mixing.
5. Intimacy of contact of ingredients.

These requirements of successful work will be discussed separately and in the order mentioned.

Purity of Ingredients.—As all the ingredients added remain to constitute the soap, it is necessary, in order to secure uniform saponification and the highest degree of brightness and freedom from discoloration, that the stock be neutral, or with the lowest percentage of free fatty acids practicable, and of good color. The quality of stock so influences the quality of the soap, that without good stock it is impossible to secure satisfactory results. The presence of free fatty acids in any considerable amount results in premature combination with alkali, causing a condition called "bunching," whereby portions of free fat and alkali are mechanically enclosed by the rapidly formed soap. Although free fatty acids enter readily into combination, their influence in producing an offensive odor in use is not thereby destroyed. The odor of the volatile fatty acids of cocoanut-oil is persistent and penetrating, and effectually precludes from the cold process any oil that has acquired any degree of rancidity.

The tallow should be of edible quality, although inferior grades are used. When of good quality, it is commonly used directly without a greater purification than that secured by settling. The quality of animal stock is shown directly in the color of the finished product, as may be seen by comparing soap made by this process from different grades of tallow. With tallow of firm body, 30 to 50 per cent of refined cottonseed-oil may be substituted for an equal weight; with tallow of soft body the percentage of cottonseed-oil should be reduced. The resulting soap will not have the initial firmness nor harden so quickly as without the use of cottonseed-oil, but the washing qualities will thereby be improved.

Caustic soda should not be of lower grade than 74°, and preferably higher for the best grades of soap. Commercial caustic of 76°

contains 98.06 per cent of the active saponifying agent; 74° caustic contains 95.48 per cent, the difference consisting of impurities, chiefly chloride and carbonate of soda. The lower the grade of caustic the more inert matter is introduced, which interferes, other things being equal, with the completeness of saponification.

Proportion of Stock and Lye.—Empirical formulæ for cold-process soap are based on the percentage of alkali absorbed by the respective ingredients and by the proportion of water it is desired to be retained by the soap. As fats and oils are varying mixtures of glycerides of different alkali absorption, and as commercial caustic soda is of varying degree of purity, accuracy of proportions of stock and lye is almost impossible. The proportions of stock and lye are likewise affected by the amount of filling added, if any—usually sodium silicate. As the different percentages of absorption of alkali of different grades by various fats and oils have been previously discussed, we need be concerned here with only the proportions obtaining in practical work.

In the following table is given the percentage of chemically pure caustic absorbed by the stock commonly used for cold-process soap, which is sufficiently accurate for the purpose of deriving formulæ:

Stock.	Per Cent NaOH.	Per Cent KOH.
Cocoanut-oil	17.5 17.7	24.6–24.8
Tallow	13.7–14.1	19.3-19.8
Cotton-oil	13.6–14.0	19.1–19.6

In order to obtain a soap of satisfactory firmness the density of lye and the proportion used should be such that the resulting soap does not contain an excessive percentage of water. It is a common practical rule that the weight of lye should be 50 per cent of the weight of stock. The density of the lye will depend upon the stock and the weight of water it is desired to retain. It happens that in pure caustic-soda solution of 40° Bé. there is twice the percentage of caustic alkali that is absorbed by 100 pounds of cocoanut-oil; so with 100 pounds of cocoanut-oil and 50 pounds of 40° Bé. caustic-soda solution, the saturation is theoretically complete. But with lye of this density best results are not secured, so a requisite amount of water must be added, or, which is equivalent, the density of the lye must be correspondingly reduced.

The derivation of cold-process formulæ cannot be better illustrated than to analyze a practical one. The following formula is one for a high-grade cold process toilet-soap of wide repute:

Cochin cocoanut-oil	600	pounds.
Caustic-soda lye 38° Bé.	315	"
Mineral soap stock	6	"
Perfume	2	"

Estimating on the basis of pure caustic-soda solution, 315 pounds of 38° Bé. lye contain 102.28 pounds of caustic soda, while 105 pounds are required to completely saponify the oil. For a mild cold-process soap this deficiency is an advantage.

If it be desired to know the weight of 36° Bé. lye required to saponify 600 pounds of cocoanut-oil, the following procedure suffices:

600 pounds cocoanut-oil × 17.52 per cent $NaOH$ = 105 pounds $NaOH$.
36° Bé. lye contains 29.93 per cent $NaOH$.
105 ÷ .2993 = 350 pounds 36° Bé. lye required.

If it be desired to know the percentage of water in a soap made from 600 pounds of cocoanut oil and 350 pounds 36° Bé. lye, the following procedure suffices:

350 lbs. 36° Bé. lye contain 105 lbs. $NaOH$.
350 " 36° Bé. lye − 105 lbs. $NaOH$ = 245 lbs. water.
245 ÷ 945 = 26 per cent water.

The percentage of sodium hydrate is absolute; the percentage of water in the finished soap is increased by increasing the volume of lye and simultaneously diminishing its density. Thus

600 lbs. cocoanut-oil require 300 lbs. 40° Bé. lye.
600 " " " 325 " 38° Bé. "
600 " " " 350 " 36° Bé. "

If it be desired to know the weight of 36° Bé. lye required to saponify 600 pounds of mixed stock containing 80 per cent tallow and 20 per cent cocoanut-oil, the following procedure suffices. Mixed stock 600 pounds:

Tallow...480 lbs×14 per cent NaOH =67.2 lbs. NaOH
Oil......120 " ×17.5 per cent NaOH=21.0 " NaOH

600 lbs. mixed stock require 88.2 lbs. NaOH
36° Bé. lye contains 29.93 per cent NaOH.
88.2 ÷ .2993 = 293 lbs. 36° Bé. lye required.

There are numerous empirical rules for the proportion of stock and lye the accuracy of which in every case should be determined by calculation before use. Some of these for unfilled soap may be mentioned. For 100 pounds cocoanut-oil 53.5 pounds 37° Bé. lye or 50 pounds 38° Bé. lye; for 50 pounds cocoanut-oil and 50 pounds tallow, 50 pounds 37° Bé. lye.

If it be desired to reduce 500 pounds of 40° Bé. lye to 36° Bé., the following procedure suffices:

40° Bé. lye contains 34.96 per cent NaOH, or 174.8 lbs. NaOH.
36° Bé. lye contains 29.93 per cent NaOH.
174.8 ÷ .2993 = 582.7 lbs. 36° Bé. lye.
582.7 − 500 = 82.7 lbs. H_2O required to be added.

For general work the most satisfactory density of the caustic lye is 36° to 38° Bé. In the preparation of concentrated caustic lye much heat is evolved by solution of the solid caustic in water. This condition must be taken into consideration in determining the density, for a hot lye of 38° Bé. will increase to about 42° Bé. when cold. Experience will determine the quantity of caustic and water required to yield a given volume of lye of the desired density. A lye indicating a density of 35° Bé. while hot will indicate about 38° Bé. when cold.

Filling.—The compound most commonly used for this purpose is sodium silicate. It may be added in any amount up to 100 per cent of the glyceride stock with advantage in so far as the appearance of the finished soap is concerned, but on aging the soap becomes stonelike in consistency and dissolves with extreme slowness with corresponding reduction of detergent power. Such soap is best applied by reducing to chips and boiling them with the articles to be cleansed. As will be indicated by the formulæ, the addition of sodium silicate in considerable amount requires the addition

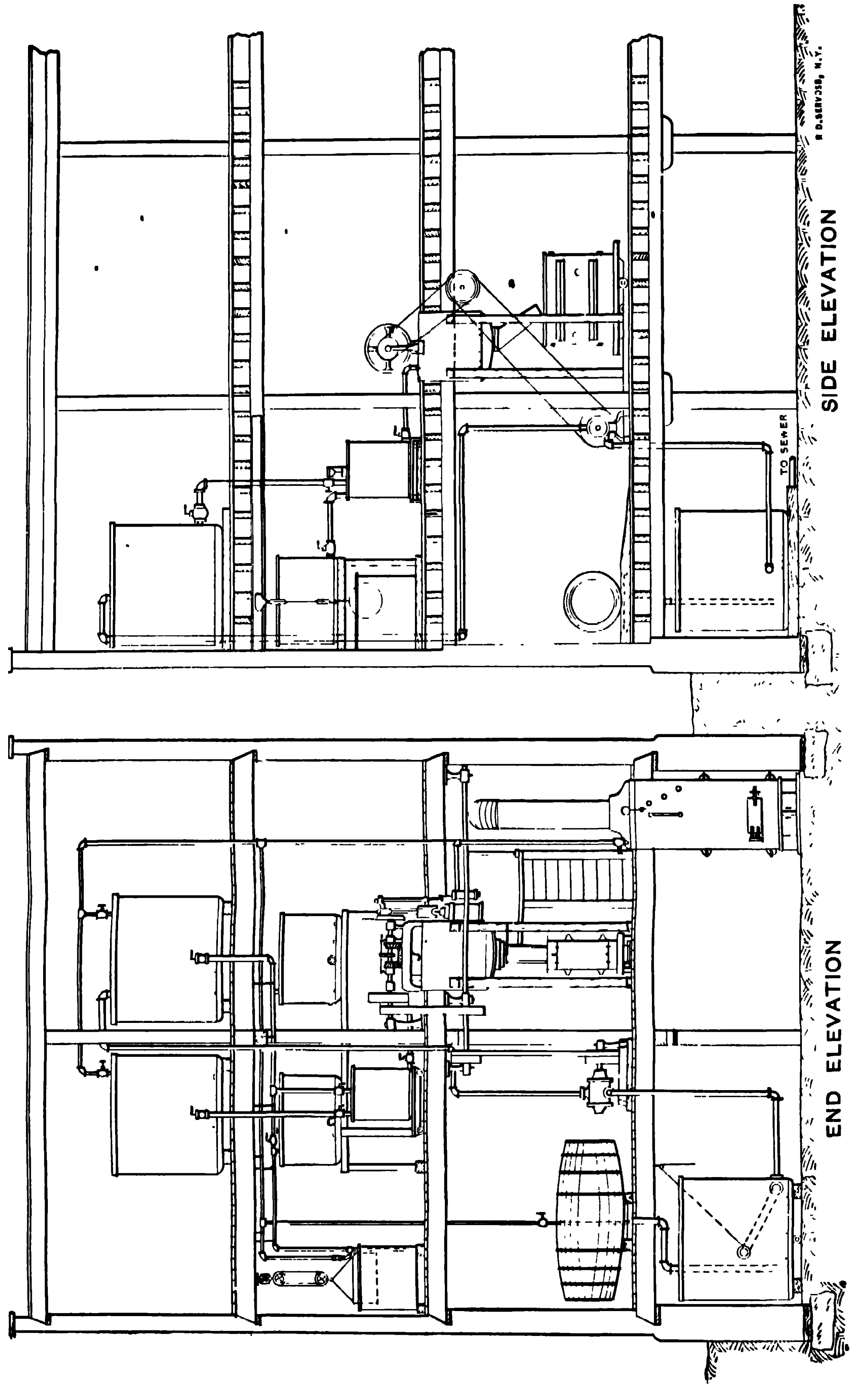


FIG. 121.—Plant for Cold-process Soap.

of more caustic lye than is called for by the stock used. The excess of caustic lye is absorbed to a large extent by the sodium silicate. In cold-process soap made from cocoanut-oil alone, salt solution, or pickle, can be incorporated to a degree limited almost only by the conscience of the manufacturer. Such soap is justly condemned by all reputable manufacturers. Carbonates of soda and potash are available as filling material, but are undesirable owing to the tendency of soap containing them to effloresce and deteriorate in appearance. Any material, however available otherwise, that sacrifices the clearness and transparency of the finished soap is likewise precluded from use as filling.

Temperature.—As it is desired to obtain only that degree of fluidity which will permit of the most intimate admixture at the lowest temperature, the most suitable temperature is determined by the character of the stock, or stock mixture, the season, quantity of filling, and lastly and most important and likewise variable, the judgment of the operator. With soap to which not over 10 per cent of filling is added, the temperature should not be less than 70°-80° Fahr. for cocoanut-oil, 110°-120° Fahr. for tallow, and 100°-110° Fahr. for a mixture of equal parts of cocoanut-oil and tallow. Temperatures higher than those stated are directly proportional to the quantity of filling and in no case should exceed the heat required to maintain the mixed ingredients in a fairly fluid state until the mass has been made thoroughly homogeneous. With an unduly high temperature, saponification ensues immediately on mixing, with the result that particles of soap enclosing fat and lye are distributed throughout the mass and are difficult to disintegrate. With stock containing fatty acids, this tendency is especially marked; hence with such stock, if used, a lower temperature than otherwise should be observed. With the frame-room at a fairly equable temperature throughout the year, no considerable change of temperature is necessary with the season beyond what is necessary to maintain the desired fluidity of the stock. The caustic lye and filling, added either successively or in admixture, should be at the temperature of the room.

Mixing.—The essential and fundamental condition preparatory to the period during which saponification takes place is thorough and rapid mixing of stock, lye, and filling. It is not desired to effect saponification during mixing more than to induce it and to introduce conditions favorable to its continuance and completion in the frame. Mixing may be done in the frame by the use of a hand-crutch, or it may be effected likewise by stirrers driven by belt, and which may be elevated and lowered as desired. The most common method is by use of power-driven crutchers.

Hand-crutched Cold-process Soap.—The following formulæ and method of operation are suited to small-scale manufacture of a filled white cold-process laundry soap. Simple and inexpensive equipment is required, the general outfit representing the minimum outlay of capital necessary in soap-manufacture.

	No. 1.		No. 2.
75 lbs.	tallow,	75 lbs.	tallow,
25 "	cocoanut-oil (Ceylon),	25 "	cocoanut-oil (Ceylon),
75 "	caustic-soda lye, 35½° B., made of 74% caustic,	70 "	caustic-soda lye, 35½° B., made of 74% caustic,
125 "	"N" silicate of soda,	100 "	"N" silicate of soda,
20 "	pearl-ash lye, 36° B.,	17 "	pearl-ash lye, 36° B.,
320 "	soap.	287 "	soap.

Weigh out the proportion of tallow and cocoanut-oil required for a frame of soap into a tight frame. Weigh out the quantity of caustic-soda lye required into a separate vessel. Also weigh out the proportion of silicate needed into another vessel; also weigh out the pearl-ash or soda-ash lye wanted, which can be mixed with the silicate.

When all is ready for mixing, the tallow and cocoanut-oil in the frame must be at a temperature of 145° to 150° Fahr. in cold weather, and 125° to 130° Fahr. in warm weather—the lye and silicate both to be at the normal temperature of the factory. When the temperature is as stated, run the lye alone into the tallow and cocoanut-oil in the frame quickly, crutching rapidly from the bottom of the frame all the time. After the lye is all in, continue crutching rapidly till the soap begins to thicken up. Now run in the silicate and pearl-ash or soda-ash lye quickly, crutching rapidly. As the silicate mixes with the soap, the whole will thin out. After the silicate and pearl-ash lye are in, continue crutching. In a few minutes the whole will gradually turn creamy. As soon as the soap becomes so thick or creamy that a mark made on the surface of the soap will remain, it is finished; take out the crutches, cover up the frame, and do not move or disturb the frame until the soap is cold. Any perfume used must be added while crutching in the silicate.

If the frame must be moved from where the soap is made, move it quickly before the silicate is added; then add the silicate at once and finish the soap as directed.

This is essentially a quick process—everything must be done quickly. For a 1000-pound frame, the lye must be run into the soap-stock in from 90 to 120 seconds— the silicate about the same. Crutching must be done quickly and the operator must reach to the bottom at each stroke—work two crutches. Never stop crutching

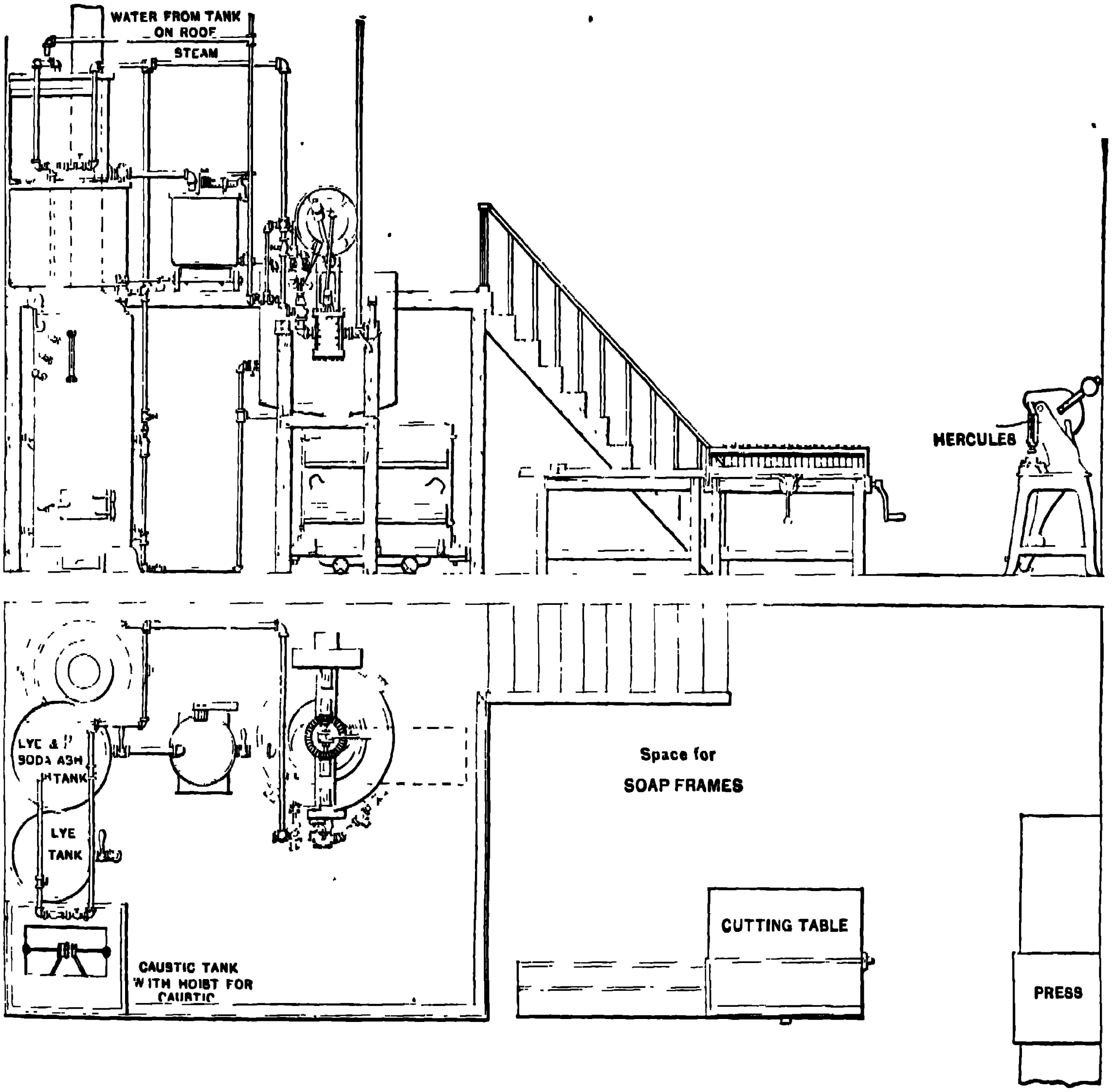


Fig. 122.—Plant for Cold-process Soap.

from start to finish. The time for making the frame (if the temperatures are right) is from 12 to 16 minutes. Great care must be taken not to crutch too long. To insure a smooth soap, stop as soon as a mark made on surface of soap will remain. Under no circumstances move or shake the finished frame of soap until cold.

Apparatus.—The factory equipment, Figs. 121 and 122, required for cold-process soap-manufacture is the simplest and least expensive. It calls first for storage-tanks for tallow and oil, and a lye tank in which caustic lye is reduced to the desired density. These ingredients may be run by gravity either directly to the crutcher, the weight used being indicated by the degree of subsidence in the storage-tanks, or first to a tank mounted on scales, where they are accurately weighed and thence to the crutcher. To facilitate handling, the silicate should be emptied from barrels into a storage-tank so situated that it may run by gravity either to the scale tank or directly to the crutcher.

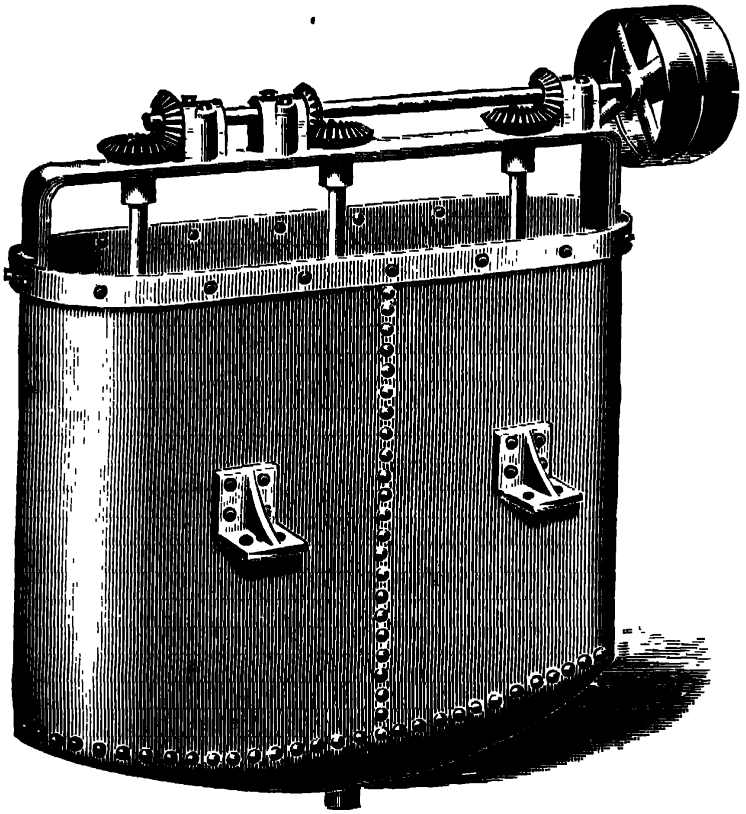


FIG. 123.—Cold-process Soap-mixer.

It is desirable that the various tanks be so situated that the materials may be handled entirely by gravity. The character and arrangement of the apparatus differ greatly in different plants. Where soap of this grade is made only occasionally the appliances of the regular soap-crutcher may satisfy every requirement. With a large

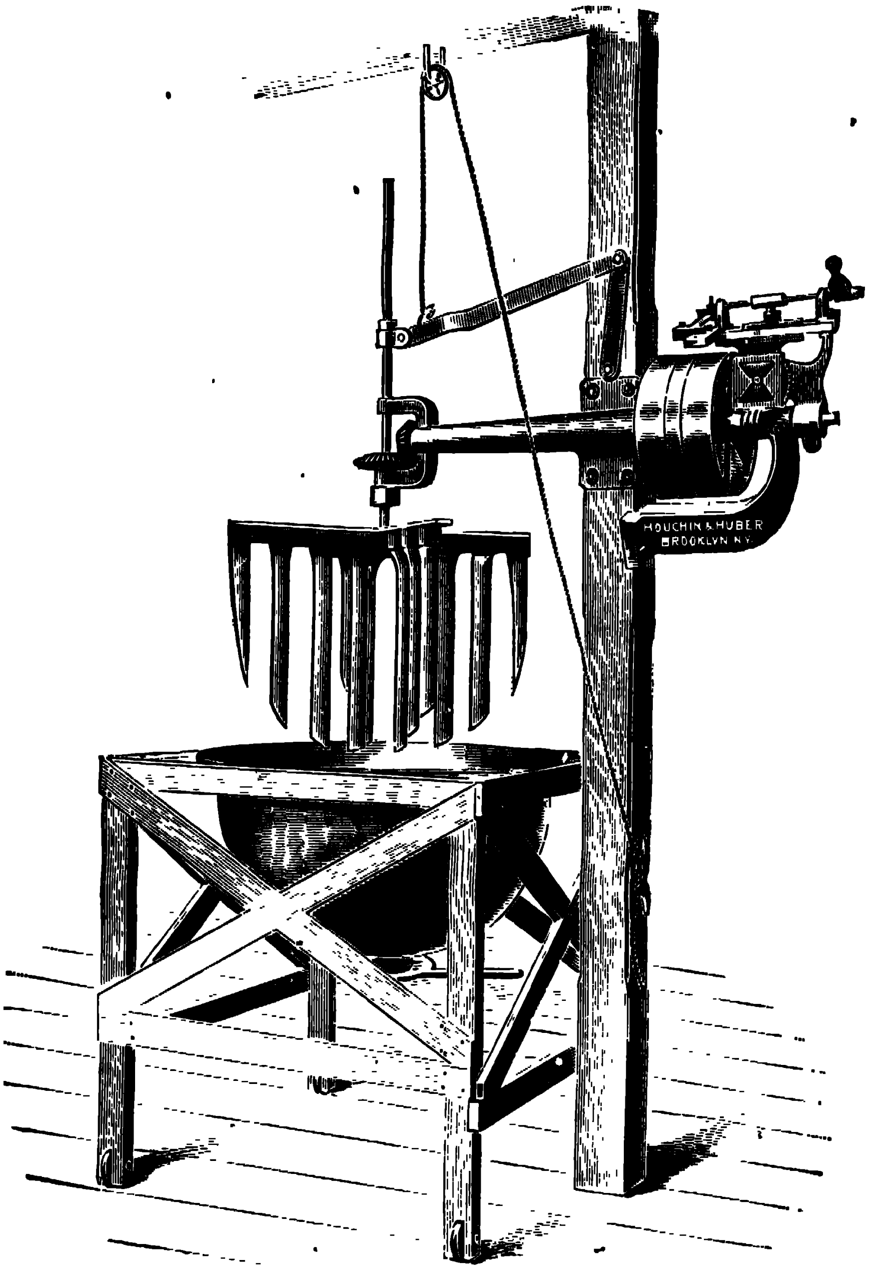


FIG. 124.—Cold-process Soap-mixer.

output of cold-process soap, the most satisfactory results are secured by providing the operator with every mechanical convenience that will facilitate the work and insure uniformity of conditions.

The regular soap-crutcher, described elsewhere, is most commonly used for mixing the ingredients. Whether this be steam-jacketed or of any particular type is largely a matter of personal preference, the chief desideratum being a thorough admixture of the contents. With a steam-jacket, however, it is often possible to correct any fault or to induce any desired thermal condition. Types of mixers for cold-process soap are shown in Figs. 123, 124, and 125 in addition to those previously described for ordinary soap crutching.

In working on a large scale, a huge crutcher, built on the Strunz type, with a capacity of 10 to 12 frames, may be used.

Referring to the types of mixers adapted for small batches shown in Figs. 124 and 125, it is seen that the agitating device may be elevated or lowered at will, thus permitting it to be raised out of the kettle or swung to one side, so that the kettle may be used as an ordinary kettle if desired.

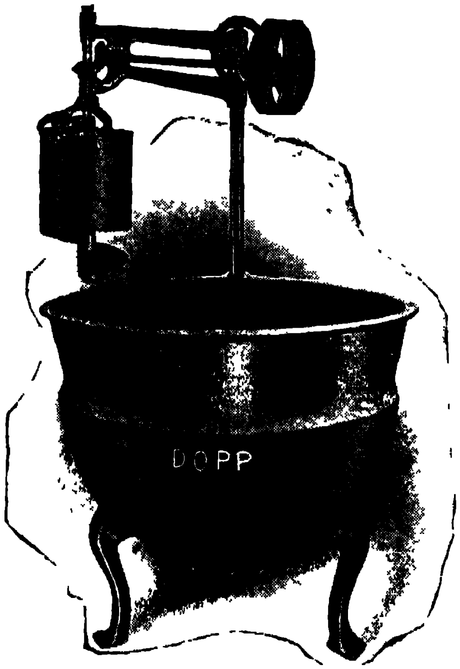


FIG. 125.—Cold-process Soap-mixer.

Mixing.—The required weight of stock for a single charge or frame having been determined either by volume or by weighing, it is run into the crutcher and the agitator started. At the same time the lye and filling are added. Whether they are mixed or added successively is determined by the experience and judgment of the operator. Crutching is continued until the mass thickens and a portion removed on a trowel appears clear and smooth and free from blotches and a mark made on the surface of the soap will remain.

The perfume may be added just previous to framing. There is much diversity of opinion and practice as to the addition of filling, which with the product of reputable manufacturers is exclusively sodium silicate. Equally good results are obtained either by adding it with the lye or just after the mixture of stock and lye has begun to thicken; however, the concensus of opinion is in favor of the former method. Experience alone must determine the proper consistency of the mass in the crutcher before framing. It is often difficult to explain the cause of unsatisfactory results, for even with the utmost apparent uniformity of operation, separation of ingredients in the frame often occurs. This separation may arise from a variety of causes, chief of which are crutching too hot or too cold, insufficient or excessive mixing, delayed framing, or cooling the framed soap too quickly.

Combination in the Frame.—Cocoanut-oil in saponifying evolves considerable heat, and it is desired that the maximum spontaneous heating should be attained, for thereby saponification is rendered more complete. After this maximum heating the soap is ready for stripping when sufficiently cool and firm. The ordinary soap frame is commonly used; to prevent excessive loss of heat by radiation, the frames may either be covered with bagging or stored in a warm place. Various types and sizes of wooden frames are used; wood, being a poor conductor, retains the natural heat. But little experience in the operation of the cold process is required to determine conditions productive of the most satisfactory results, and frequently these results, both good and bad, when obtained, baffle explanation.

Laundry Chipped Soap.—The cold process is used to a large extent for the manufacture of soap to be chipped for use in steam laundries. The following formula is for a neutral chip soap:

Tallow	294	pounds.
Cocoanut-oil	70	"
Silicate of soda	184	"
36° Bé. caustic, 74°	200	"

The following formula is for a cheaper grade containing an excess of strength:

Tallow	310	pounds.
Cocoanut-oil	20	"
28° Bé. caustic, 74°	350	"
Silicate of soda	36	"

The following formula provides for 100 per cent of silicate of soda on the basis of the glyceride stock and produces an excellent soap of this grade:

Tallow	228	pounds.
Cocoanut-oil	56	"
36° Bé. caustic, 74°	216	"
Silicate of soda	284	"

For a white laundry soap or soap chips the following formula has been found to give good satisfaction:

Prime tallow	235½	pounds.
Ceylon cocoanut-oil	50	"
Silicate of soda	137	"
37° Bé. caustic	183½	"
Mineral soap-stock	20	"

Temperature 130° Fahr. for tallow and oil.

The proportion of cocoanut oil may be increased if desired and the amount of tallow used decreased.

Silicate and lye are mixed together.

An excellent base for soap chips can be made in accordance with the following formula:

Tallow	500	pounds.
Cottonseed-oil	50	"
Cocoanut-oil	50	"
Caustic-soda lye, 35° Bé	350	"
Caustic-potash lye, 35° Bé	40	"
"N" silicate	300	"

Melt the stock and add it to crutcher at 135° Fahr. While in agitation, add lye and silicate simultaneously. After the soap has thickened and appears smooth and lustrous on the paddle, it is ready for framing. Grease may be substituted for a portion of the tallow, but only at a sacrifice of bright color of the chips.

For a white soap the following formula is recommended:

Tallow	95	pounds.
Cocoanut-oil	5	"
35° Bé. caustic	65	"
Silicate of soda	50	"

Tallow and oil are melted and mixed at 112°–118° Fahr. in summer and at 120°–125° Fahr. in winter. In summer lye and silicate of soda may be mixed at room temperature, but in winter it is recommended that the mixture be heated to 130° Fahr. Add lye and filling to the mixed stock while the latter is in a state of agitation and continue crutching until the mass becomes firm enough to retain an impression. The large proportion of silicate of soda in this formula serves to absorb the excess strength. The formula may be modified to produce a cheaper article by substituting white grease in judicious proportions for an equivalent weight of tallow.

Chipping Soap.—After the maximum heating has been attained cold-process soap is ready to strip as soon as firm enough to stand. For chipping the soap is slabbed and cut into bars in lengths equal to the width of the frame. For this shape the second cutting-head of the cutting-table answers. After drying the bars are ready to be chipped. The principle of the soap chipper is seen in the type shown in Fig. 126. The knives are adjustable, so that the soap may be reduced to chips of any desired thickness. In Fig. 127 is shown a chipper with knives arranged horizontally and designed for the chipping of long bars which are placed by the attendant in the cylindrical hopper surmounting the knives. In Fig. 128 is shown in cross section a continuous chipping and drying device adapted to large-scale production.

Attention is directed to this device for the reason that it represents an intermediate stage in the simplification of the manufacturing process which finds its present culmination in the continuous crusher and dryer described on page 436. Attention at this place is directed to the latter device for the reason that its use represents the highest possible economy in the manufacture of chip soap.

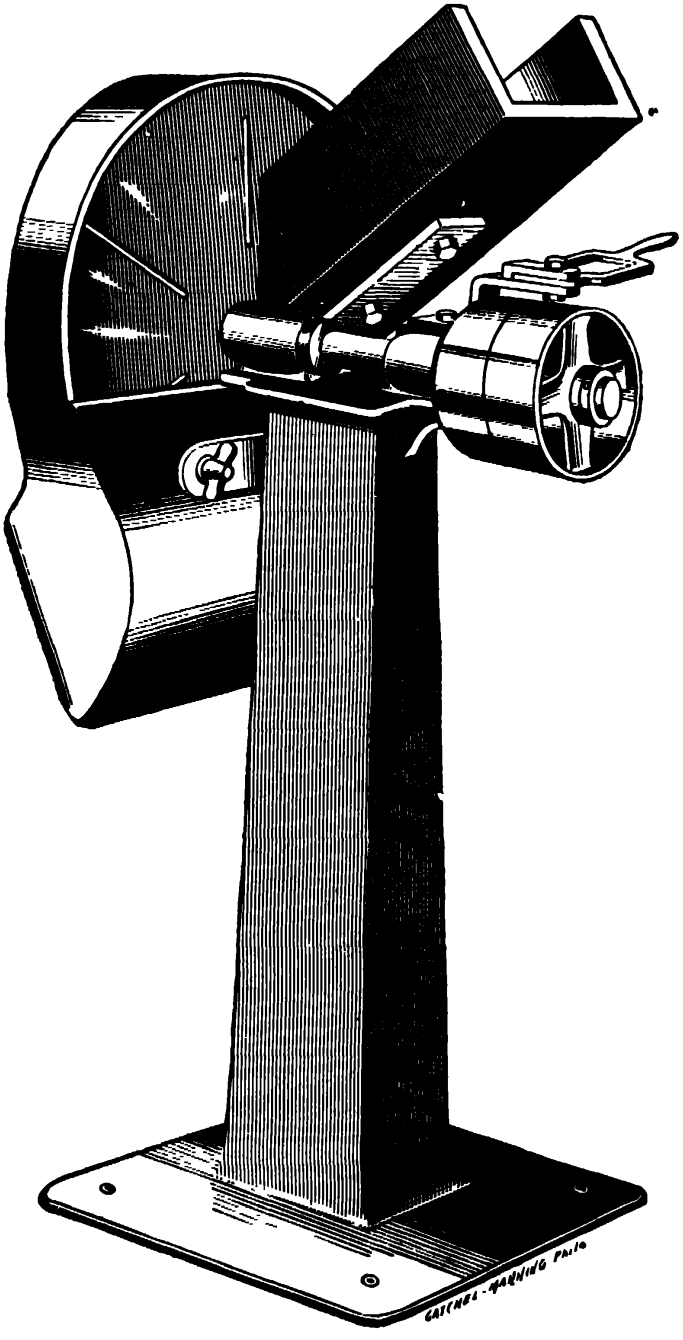


FIG. 126.—Type of Soap-chipper.

The chipper and continuous-drying device shown in principle in Fig. 128 is seen to consist essentially of a series of horizontal belt carriers made of wire netting enclosed in a case into which hot air is projected, as shown on the right, for drying the chipped soap conveyed thereon. On the left is shown a smaller fan for introducing cold air for cooling the chipped soap so that it may be ready for packing into barrels when discharged from the case. In this explanatory diagram the chipper surmounts the drying-chamber into, and on to the topmost carrier of, which it discharges the chips. The chips are carried from left to right and, vice versa, from above downward and undergo a continuous drying process, meeting finally at the bottom a draft of cold air which effectually cools them and counteracts the tendency to sweat when tightly packed in barrels. The drying-chamber and movable parts are constructed throughout of metal.

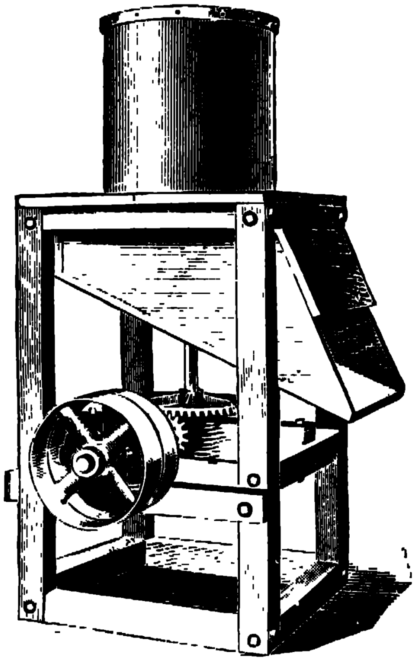


FIG. 127.—Slab-chipper.

Cold-process Soap from Fatty Acids.—Place the caustic lye in the crutcher. Set the crutcher in motion and then add the melted fatty acids in a fairly rapid stream, so that the entire charge of the fatty acids will be added in the course of two or three minutes. A partial union of fatty acids and caustic takes place at once, but the soap remains thin and with a slightly open appearance for from 5 to 10 minutes, after which it thickens and looks close and smooth. If filling is to be used it should be added at this point if the temperature does not exceed 150° Fahr. The length of time the soap should be crutched after thickening and having the filler well mixed through, if a filled soap, will depend on the kind of soap desired; such other variable circumstances as speed of crutcher, etc., can be best determined by a few runs in each particular case. As a general thing the soap should be dropped from 15 to 30 minutes after all the fatty acids have been introduced. A jacketed crutcher should be used and the temperature of the lye and fatty acids, as well as that of the finished soap, will depend upon the kind of soap being made, and can also best be determined by a few runs in each case. From 140 to 160° Fahr. will probably be

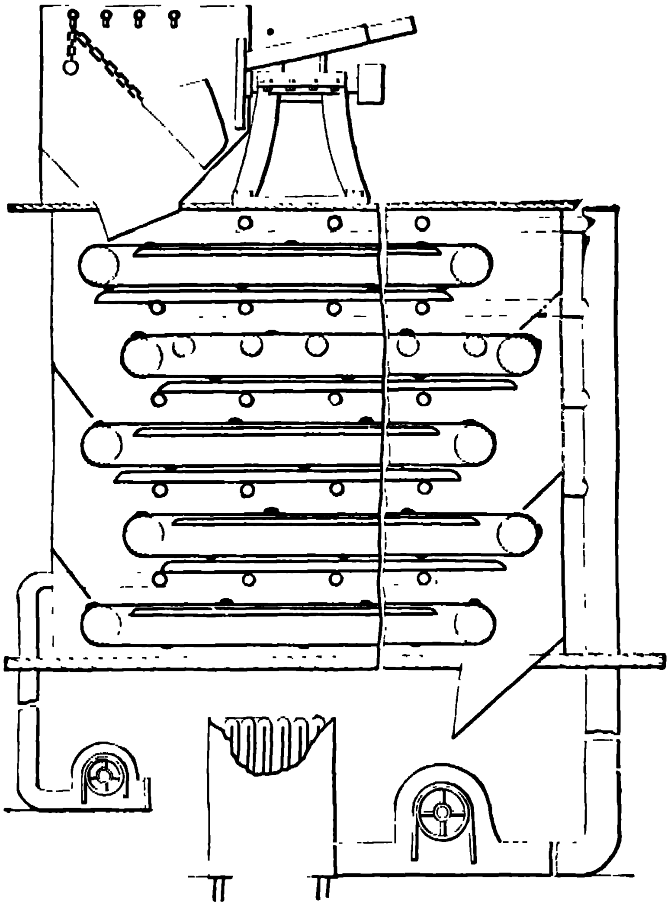


FIG. 128. Chipper and Continuous-drying Device.

the best temperature at which to drop the finished soap as a general thing. This would require a somewhat lower temperature of the lye and stock before mixing. If rosin is used in such a soap it should be melted in the fat mixture before running into the crutcher.

Cold-process Toilet-soap.—The incomplete combination of the ingredients of cold-process soap, whereby free fat and free alkali exist side by side, unfit this process for the production of high-grade toilet-soap, yet much soap for toilet purposes is made by the cold process.

For soap of this quality only Cochin cocoanut-oil and edible tallow should be used, thereby securing the highest practicable neutrality; also, lye made from high-grade caustic is necessary. The same procedure is followed as with cold-process soap for industrial and domestic use. The better the stock and the greater the care used, the finer is the product. For a pure cocoanut-oil soap the general rule, that caustic lye of 38° Bé. to the extent of 50 per cent of the weight of oil be used, should be followed. As has been already shown, this proportion leaves an excess of free oil if all the caustic be absorbed. This will yield a smooth, semi-transparent, profusely lathering soap, but which invariably will show rancidity on aging.

By substituting tallow for a portion of the cocoanut-oil in accordance with the following formula,

Tallow	80	pounds
Cocoanut-oil	120	"
Caustic lye, 37° Bé.	100	"

the washing properties of the soap will be modified to advantage. The perfume should be added just before framing if a power crutcher is used; if made in small wooden frames, at the last stage of the crutching and thoroughly worked through. If the soap is colored completely, the dye should be mixed with the stock, with which all organic coloring-matters suitable for soap of this quality will readily amalgamate. The dye should be thoroughly reduced by grinding in water or oil and strained to avoid introducing any particles whereby specks or blotches would be produced. To produce an artificial mottle or marbled appearance, the dye should be added in requisite amount after the incorporation of the perfume and worked through only to the degree that it is disseminated throughout the mass of soap in streaks. For this purpose water-soluble dyes should be avoided, as they disseminate too readily. Insoluble mineral dyes are preferable, as Venetian red, the various ultramarines, chrome green, etc. All dyes of whatever origin acted upon by alkali should be avoided. For coloring soap many dyes—fast, harmless, and alkali-proof—of coal-tar origin are available. In using dyes, care must be observed to avoid such an excess as would stain the hands or discolor the lather on using the product.

The production of so-called tar soap by the cold process, characterized by a distinctive odor and a brown color when fresh and black on aging, is effected by amalgamating with the stock before the addition of lye the requisite amount of pine tar.

Although cold-process toilet-soap enjoys a certain sale, its use has been largely displaced by the more attractive, cheaper, neutral, and more generally satisfactory milled variety.

Utilization of Scraps from Cold-process Soap.—The utilization of scrap is a problem varying in importance with the volume of cold-process soap made and with the variety of soap-manufacturing processes carried on. Where white floating soap is made, white cold-process scraps may be added to the kettle charge of boiled soap; or they may be added to the kettle charge of rosined soap and rosined up to the required proportion. Where the scraps are deeply colored, as with tar soap, they may be allowed to accumulate until there is sufficient amount to remelt alone and frame. For this purpose an ordinary remelter, elsewhere described, may be used. This procedure, however, is troublesome, as the discoloration and odor affect subsequent additions of different soap unless the remelter is thoroughly cleaned. Again, the texture of soap thus treated is not as desirable as that of the original soap.

The utilization of white cold process scraps presents no difficulty. They may be remelted alone in a steam-jacketed kettle provided with an open steam-pipe, either with or without the addition of fresh stock and lye, and after crutching framed for use as a base for chip soap. The following formula provides for a frame charge of scrap, fresh stock, and lye and silicate. The proportions of tallow, cocoanut-oil, lye, and silicate may be the same as for the original soap, and may be varied in quantity in accordance with the amount of scrap.

White cold-process scrap.	750	pounds
Tallow.	114	"
Cocoanut oil.	28	"
36° Bé. caustic.	125	"
Silicate.	125	"

Final Considerations.—Cold-process soap has a characteristic, compact texture, and retains its shape longer and deteriorates less in appearance on aging than does a settled soap. By the use of a proportion of caustic potash in saponification a marked improvement in the product is possible, the soap being milder, of finer texture, and of superior general appearance. As more caustic potash than caustic soda is required for saponification, a given quantity of the latter may be transformed into an equivalent amount of the former by multiplying the absolute amount of caustic soda by 1.4, this factor expressing the ratio of the combining weights of the two alkalis.

The cold process possesses numerous advantages, chief of which are the low cost and simplicity of manufacturing plant, whereby fixed charges become almost inconsiderable with steady output of product, ease and rapidity of manufacture, whereby the volume of output may be adjusted quickly to the demand, and the higher degree of sophistication and greater yield than are possible with hard soaps yielded by either the semi-boiled or settled processes. The only disadvantage of the cold process is the invariable incompleteness of saponification, which restricts an almost ideally efficient manufacturing process to a very limited field. Where cold-process soap is used in considerable amount, the advisability of recovering the glycerin which is retained by the soap will always warrant consideration. The recovery of the glycerin, however, is possible only with a change of the process of manufacture.

Modified Cold Process.—As the manufacture of soap by the cold process possesses many advantages from the standpoint of economy of production, any modification of the process whereby mixtures of fats and oils and alkali are made to combine at a higher temperature would not only extend its field of usefulness but would permit of a more satisfactory product. A change of this character, although it does not arise to the distinction of a modification, for the essential principles and procedure remain practically the same, lies in effecting combination at a higher temperature and under conditions that differ with different kinds of soap. In the cold process, as commonly understood, it is endeavored to effect in the crutcher merely a thorough incorporation of the ingredients, and when done, the soap is framed and saponification with spontaneous heating is allowed to ensue. Under the conditions of the change stated the original temperature of the stock is from 140° to 160° Fahr., and the mass is retained in the crutcher and manipulated until after the period of spontaneous heating which ensues in the cold process in the frame. While more time is consumed in the preparation of each frame of soap, a certain satisfaction accrues to the operator in being able to control the reaction at will, instead of allowing it to proceed with uncertain results in the frame. The various cold-process formulæ are applicable under the conditions of higher temperature obtaining in this method.

Toilet-soap.—The following formula and procedure have been recommended for a cheap toilet-soap.

Formula.

Tallow, edible..........	450 pounds	Heat to 140° Fahr.
Cochin cocoanut-oil......	50 "	
36° Bé. caustic, 74°......	300 "	

Procedure.—Add the tallow and cocoanut-oil to a jacketed crutcher and heat the mixture to 140° Fahr. Add 36° Bé. lye, with crutching, and as soon as the mass begins to thicken, cover the crutching to prevent loss of heat and allow it to stand for one hour; then crutch the mass for ten minutes and again cover, allowing the soap to stand two hours longer, during which time saponification ensues. In the meantime make the following compound: Heat up 30 pounds of oil to 120° Fahr. and thoroughly mix 30 pounds of potash solution 36° Bé. and 30 pounds of soda-ash solution 36° Bé., after which add a sugar solution made from 9 pounds of granulated sugar and 13 pounds of hot water. After thorough admixture of this compound, it is added to the crutcher and thoroughly incorporated. Any desired perfume may now be added and crutching continued until the soap appears smooth and thick. The soap is then ready to frame with the usual precautions. The filling may be modified or omitted as desired. The following formulæ may be used for the same purpose and with the same general treatment:

No. 1.	Pounds.	No. 2.	Pounds.
Tallow....................	440	Tallow....................	350
Cocoanut-oil..............	60	Cocoanut-oil..............	200
34° Bé. caustic-soda lye....	220	Castor-oil................	50
30° Bé. caustic-potash lye...	60	36° Bé. caustic-soda lye.....	300

Use of Rosin.—The natural consistency of rosin does not adapt it for use directly in the cold process; neither is it suitable when melted, for its presence in admixture with glyceride stock exercises the same influence as an excessive amount of free fatty acids in a fat or oil. By the complete saturation of rosin with alkali in the soap-kettle, it is made available for use as "rosin compound" in the modified cold process for the manufacture of cheap laundry soap. The rosin compound may be made by the direct saturation of rosin with 20° Bé. lye. When the rosin is completely killed the rosin soap may be allowed to settle and used as required. By combining a certain proportion of tallow or grease with the rosin the whole may be grained and subsequently closed, and thereby to a degree purified. Any quantity of stock may be prepared in this manner and used as desired.

In the manufacture of laundry soap of this character the property of starch to form a gelatinous mass when boiled with caustic lye may be taken advantage of to form a binding agent. With the use of this agent and sodium silicate and a stock base made of rosin compound, fresh stock, and lye, laundry of good initial appearance and high detergency may be made at low cost. The use of a large amount of alkaline filling renders the presence of a binding agent necessary. A powerful crutcher should be used. In carrying out the process the required weight of stock and rosin compound are added to the crutcher. The caustic lye and silicate are then added and thoroughly mixed. Separation of ingredients is checked by the addition of starch or mineral soap-stock.

Tar Soap.—Tar soap takes its name from the addition of pine tar, which is obtained by the destructive distillation of pine wood. It possesses marked disinfectant properties, which are generally ascribed to the soap containing it and which are due to the presence of phenol and its homologues, cresol and guaiacol. Organic acids, notably acetic, are present in considerable amount. The compounds mentioned combine readily with alkali, which explains the alkali-absorption property of this material as observed in the kettle. It is generally required of a tar soap that it be of dark color, smooth, of glossy finish, of tough texture and pronounced odor of pine tar and of quick-lathering properties. The amount of pine tar used is extremely variable, ranging from 5 to 25 per cent in the more popular brands. The freshly made soap is brown, even if it contain 25 per cent of tar, the rapidity with which it turns black on exposure depending upon the proportion of tar used. The curative properties of tar soap, as well as those generally attributed to the so-called medicated soaps, will receive separate discussion on a subsequent page. Tar soap may be made by the cold process, or, with much greater ease, by the simple addition of the tar, neutralized with alkali, during crutching, to either a semi-boiled or grained soap. With the use of the acid, unneutralized tar it is sometimes the practice to kill it with the stock in the kettle in the manufacture of straight semi-boiled soap, to be sold as such or to be used as a base for milled soap. Cocoanut-oil imparts the quick-lathering properties and may be used in any proportion from 25 to 50 per cent with tallow. The advantage of the use of rosin in soap of this character readily suggests itself. Such soap is a good receptacle for soap scraps of any quality or color, although the proportion worked up must not be sufficient to modify the character of the soap desired. The use of sodium silicate is recommended; soda-ash must be used sparingly, if at all, to avoid efflorescence.

The intensity of color and odor of tar soap may be varied by either varying the proportion of tar used or by using any desired quantity of the soluble aniline blacks suitable for this purpose. The alkaline absorption of pine tar, while variable, is considerable.

Formula.

Cocoanut-oil	300	pounds
Tallow	300	"
Pine tar	80	"
36° Bé caustic	375	"
Silicate of soda	40	"
Pearl-ash lye, 36° Bé	10	"

With scraps on hand 200 pounds may be added to the above mixture.

Procedure.—Heat the mixed stock and pine tar to 180° Fahr. When scraps are used, add them to this mixture, and when melted, add to the mixture, already in the crutcher, the mixed lye and silicate. Crutch the mass for about five minutes and allow it to stand for an hour, then repeat the crutching until the whole is combined. Add the potash lye and crutch until the soap becomes smooth and thick, when it is ready to frame.

While a fairly satisfactory tar soap can be made by the cold process, the best results with this grade of soap are obtained by milling. The preparation of the soap base and the milling process are described later. The incorporation of tar in the crutching process involves no difficulty other than the necessity for the thorough cleansing of the crutcher before using it for other purposes. The proportion of tar used depends upon the intensity of odor and color desired; the permanency of the latter characteristic may be insured by the use of soluble aniline blacks as stated. The proportion of tar or color in no case should be so excessive as to strongly color the lather. The preparation of colorless products of the destructive distillation of the pine possessing the characteristic odor admit of a "white" tar soap.

A writer in the *American Soap Journal* gives the following directions for the manufacture of tar soap by the semi boiled process:

"To make a good article of this soap is not such an easy matter as would appear at first sight. A great many of the tar soaps in the market are milled soaps, and as such are beyond our means to go to such an expense for machinery.

"Now, if we can turn out a soap that will lather quick, look dark, glossy, and smooth and be tough, so that it will stand pressing into the round bar so generally adopted for this kind of soap, we have got a boiled soap that the majority of mankind can't tell from a milled soap, and something that will give the best satisfaction in the using of it.

"It is not so much the amount of tar that gives it the dark, glossy look so much desired as the finish. Even if large quantities of tar are used, if not finished right, the soap will look brown in color. The way that I have found it best to work is like this: I will take (say for one frame) 300 pounds of tallow and 300 pounds of cocoanut-oil, or 400 pounds of tallow and 200 pounds of cocoanut (less than this amount of cocoa I don't approve of). Put in small kettle (with dry coil in it) all the tallow and about half of the cocoanut-oil. Then commence giving it lye at 8° to 10° Bé. until thoroughly joined. Then gradually increase the strength to 25°-30° Bé. When saponified, it will be open and taste sharp. Continue the boiling for a few minutes. Then add about 6 gallons of pine tar. Having previously melted the cocoanut-oil left out, commence feeding it slowly. As it takes up the strength the soap will assume a smooth, dark, and glossy appearance. Keep adding the melted oil slowly, watching closely the soap for signs of thickening up. When the strength is about all taken up and the soap tough, so that it won't slip off the trowel but drop in a thick mass like thick molasses, it is ready to dip out into the frame. (If by any chance it should thicken up before you are ready, a little strong lye 25°-28° Bé. will thin it out, which strength must be taken out again.) It should thicken up in a short time in the frame; if it don't you will have to crutch it until it is thick, so that it won't settle any nigre. Tar soap made in this way will give the best satisfaction. It will be smooth to the feel, glossy, and almost black in color, and will answer for all the purposes of milled soap, with not half the trouble to make it. It can be made in smaller or larger quantities if desired, but one frame is what I have found best for our purposes. Good No. 2 tallow can be used if desired, also the Ceylon cocoanut-oil; cold-made soap scraps, red and white castile scraps, and cocoanut castile scraps can all be worked in by putting them in early so that they have time to melt thoroughly before it is closed up. But don't use too much silicate."

Transparent Soap.—The solubility of soap in alcohol and the transparency of the residue on the evaporation of the solvent were early taken advantage of in the manufacture of transparent soap, and for a long time the solution of well-made settled soap in alcohol remained the essential procedure for the manufacture of this grade of soap. This procedure yields the best results, but has long since been discarded owing to the expense, save in a few isolated cases of well-established brands. In the cheaper processes used at the present time, alcohol may be simply added to the soap, made either by the cold- or modified cold-process, at some particular stage of the saponification. The ready solubility of soap in alcohol facilitates saponification. It was found that glycerin conduced to transparency, which in addition to its emollient properties led to its substitution for a portion of the alcohol. Sugar solution in time partially and finally completely displaced glycerin, and in the course of the cheapening of the process it was found that satisfactory transparency could be produced by the use of sugar solution alone without the aid of alcohol or glycerin. For producing the transparent effect it is generally believed that sugar solution is more efficient than glycerin, which, unless used with care, tends to make the soap sweat through its affinity for moisture.

Ingredients.—Transparent soap demands the best and purest material and the complete saponification of the stock. With the exception of castor oil, stock for transparent soap does not differ from that used for high-grade milled and floating soaps. The marked solubility of castor-oil and of its sodium salts in alcohol adapts it well as an ingredient for soap of this character, and with its use less of the substances conducive to transparency are required. Caustic-soda lye should be prepared from high-grade commercial caustic and be well settled. Filling material, whether glycerin, sugar solution, or soda-ash solution, must be clear and colorless, and for light-colored soap care must be observed that the sugar solution is not darkened by undue exposure to heat. To produce a clear and transparent effect all insoluble matter must be eliminated; this necessitates use of the best and purest material. Cloudiness to a greater or less degree generally ensues on aging, even in the most skilfully made transparent soap. Its development in the finished soap may be hastened by incomplete saponification of the stock, by crystallization of alkaline salts used for the purpose of filling and to overcome the softening effects of sugar and glycerin, or by mineral impurities.

The combination of glyceride stock for transparent soap illustrates well the blending of properties of individual fats and oil. Castor-oil imparts transparency to the finished product, cocoanut-oil ready solubility, and tallow firmness. These properties, however, are greatly modified by the proportion of filling used.

Processes.—While the procedure of manufacture by the cheaper methods is that of the modified cold process, three general classes of processes may be distinguished, according to the nature of the material used to produce transparency, as follows: (1) Soap made with alcohol and glycerin; (2) soap made with alcohol and sugar; (3) and soap made without alcohol. For these three general classes the same stock composition may be used which will comprise in a broad way equal parts of cocoanut-oil and tallow with sufficient castor-oil to facilitate combination and to produce the desired transparency. Whatever variation may be made in the proportion of tallow and oil, the proportion of caustic-soda lye, usually 38° Bé., to the total weight of cocoanut-oil, tallow, and castor-oil, remains practically 50 per cent. The following formulæ will serve to indicate the general variation in the proportion of ingredients in transparent soap of the three classes. The proportions throughout are by weight.

(1) *Transparent Soap with Alcohol and Glycerin.*—The following formulæ of this class produce the best and most expensive soap, but are of limited application owing to the high cost. The large proportion of alcohol and glycerin is sufficient to produce transparency without the aid of castor-oil.

Ingredients.	I	II.
	Lbs.	Lbs.
Cochin cocoanut-oil	100	120
Edible tallow or its equivalent	100	60
Caustic lye, 38° Bé.	100	90
Alcohol, 95 per cent	80	60
Glycerin	40	60

(2) *Transparent Soap with Alcohol and Sugar.*

Ingredients	I.	II.	III.	IV.
	Lbs.	Lbs.	Lbs.	Lbs.
Cochin cocoanut-oil	70	70	100	100
Edible tallow	40	50	100	100
Castor-oil	30	20	30	30
Caustic lye, 38° Bé.	70	70	115	115
Alcohol, 95 per cent	40	40	100	100
Sugar *in*	60	40	25	60
Water	60	30	40	75

The proportion of soda-ash solution, 36° Bé., required to harden the soap depends upon conditions and the judgment of the operator.

(3) *Transparent Soap Without Alcohol.*—As the proportions of alcohol and glycerin are reduced to entire removal, the proportions of castor-oil and sugar solution are necessarily increased to effect the same transparency in the product.

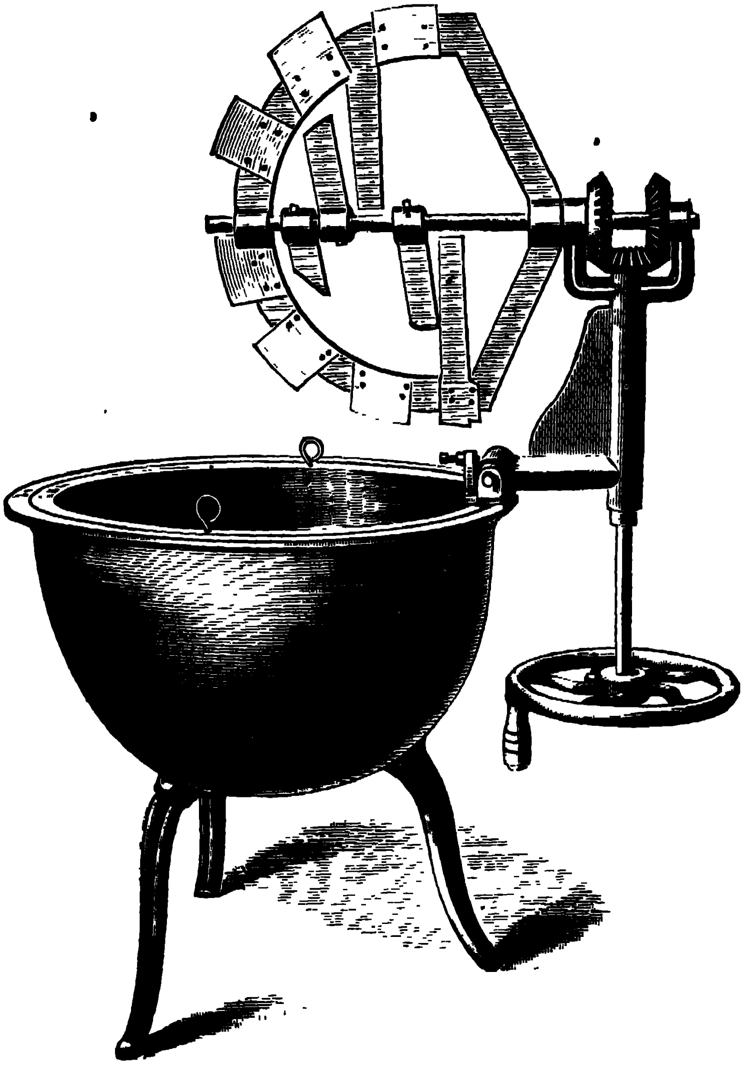


FIG. 129.—Water-bath Mixer for Transparent Soap.

Ingredients.			III.
	Lbs.	Lbs.	Lbs.
Cochin cocoanut-oil......	74	26	130
Edible tallow...........	60	30	147
Castor-oil....	76	37·5	180
Caustic lye, 38° Bé......	108	56 (35° Bé.)	261 (34½°)
Sugar *in*...............	60	25	130
Water..................	64	25	130
Soda-ash solution, 36° Bé.	30	10	25

Procedure.—The mixed stock is melted, settled, weighed, and carefully strained into a steam-jacketed crutcher and heated up to about 130° Fahr. The requisite amount of caustic lye at the temperature of the room and of the density that has been found to produce the best results, not varying much, however, from 38° Bé., is added and thoroughly mixed. This procedure is after the general manner of working cold-process soap. The mixture is now allowed to stand for an hour or more, the crutcher being covered to retain heat, during which time the mass undergoes the spontaneous heating incident to saponification, the temperature rising to 180° Fahr. or more. The progress of spontaneous heating should be carefully observed by means of a thermometer.

If the formula includes glycerin it should be added and mixed with the stock before the addition of caustic lye. If, after thorough mixing of the lye, saponification is for any reason retarded, steam should be carefully admitted to the jacket and the mass vigorously crutched until combination is induced. Steam is then shut off. If the formula includes alcohol it should be added after the addition and mixing of the caustic lye and previous to allowing the mixture to stand for spontaneous heating.

In the meantime the sugar and soda-ash are dissolved in the required amount of water and the solution heated to about 180° Fahr. At the expiration of the period of spontaneous heating, determined by the thermometer indicating a permanent, maximum temperature, crutching is begun and the solution prepared as before described is slowly added. The mass in the crutcher is now reduced to a thin, syrupy consistency. Any scrap on hand to be worked over may be added at this time. When the scrap has thoroughly amalgamated and the mass is homogeneous, crutching is discontinued and the crutcher covered and allowed to stand for at least two hours, during which time a froth, or fob, rises to the surface, which should be removed. Samples should now be taken. If conditions have been satisfactory, the samples when cold should cut up into perfectly transparent flakes, uniform in color and have no excessive alkaline taste. If the sample is firm but turbid, more sugar-and-soda solution is required, which should be added carefully from time to time, with continued crutching until the desired transparency is attained. Extreme care should be exercised that an excess be not added. If the sample has the desired transparency, but lacks firmness and has a neutral taste, more caustic lye should be added, with the same precautions against excess. If the sample lacks firmness, but has the desired transparency and alkaline taste, an excess of water is present, which may be corrected by the addition of dry ash. An excess of alkaline carbonate in the finished soap induces efflorescence. If from an excess of caustic lye or carbonate the soap should tend to separate or grain, cocoanut- or castor-oil should be carefully added until the soap is closed. Should the sample cool quickly and set stiff more water is needed. Incomplete saponification, indicated by either a sharp taste or soft and greasy feel, or both, can be corrected only after prolonged heating and crutching, and this always at a possible sacrifice of color. Saponification should be complete before the sugar solution is incorporated; any subsequent heating to correct a deficiency in this respect deteriorates more or less the color of the finished soap. With soap dyed a dark color this danger is of less consequence. With conditions satisfactory the soap is cooled to about 140° Fahr. and the dye added, dissolved first in hot water and strained, and thoroughly incorporated with the soap. Crutching is continued until the soap is cooled to about 130° Fahr., when perfume is added. The mass is now ready to frame. This should be done as quickly as possible, the soap being strained as it runs into the frame. To reduce the percentage of scrap to a minimum and to obtain better results in pressing, the fluid soap may be run into elongated tins corresponding in cross-section to that of the dye. When cool the elongated bars may be easily removed and reduced by cutting to the desired thickness.

After the soap is framed it should be allowed to cool quickly and to stand as long as possible before and after stripping, and after cutting prolonged drying under proper conditions before pressing tends to improve the appearance of the soap. In cutting, allowance should be made for shrinkage during drying, and also the cake should be cut to conform as closely as possible to the dimensions of the die to avoid cracking during pressing.

SEMI-BOILED SOAP.

Semi-boiled Defined.—The term "semi-boiled" refers to a soap that has not been grained and that differs from a cold-process soap in that the combination of fat and alkali has been effected at a higher temperature and under conditions that admit of complete saponification. The term refers to the process as well as to the product made by the process. The term "semi" has reference in a general way to the fact that soap of this class is not subjected to as prolonged, but not necessarily to a less thorough, boiling as occurs in the manufacture of grained, settled soap. Semi-boiled soap partakes of the general character of settled soap inasmuch as on the final change the soap is left in such a condition that a degree of purification is effected by the settling of a nigre. By basing a classification on the use of salt as a graining agent a sharp, practical distinction between the two classes of boiled soap may be made. As there has been no separation of the material by graining, the vessel in which the soap is made will contain on the completion of the process all the ingredients originally added to it. The process is carried out in steam-heated kettles, the skill and experience of the soap-boiler being depended upon to determine the satisfactory combination of the ingredients without accurate regard to the relative proportions or weights of materials used.

Since in the semi-boiled process combination is effected by the aid of steam, the capacity of the steam-making plant must be greater than what is required for the operation of the simple machinery used in the cold process. A greater outlay is required for manufacturing equipment, which is more intricate than that required for the cold process, in the necessity of a boiler-plant of ample capacity, piping and necessary appurtenances for the proper handling of steam, water, stock, lye and soap, riveted sheet-steel kettles of varying capacity, provided with open and closed steam-coils for heating and suitable pipe connections for the discharge of their contents, and pumps for the transference of material in liquid form Otherwise the essential equipment is the same as for the cold process.

Of the two processes of boiling soap, viz., the semi-boiled and grained, or settled process, the former is a cheap and quick method and is especially adapted for use in the manufacture of soap that can be prepared more economically and with equal satisfaction than by the settled process. Although all the materials added to the kettle remain to constitute the soap, the degree of purification affected by the settling of a nigre, if desired, allows more latitude in the quality of stock than does the cold process. The process at best, however, is very limited in its application.

All soap made by the semi-boiled process can be made with equally satisfactory results by the settled process with the great advantage that the glycerin in the stock may be recovered. For the best results by the semi-boiled process, the stock should be of good quality, which is to say that the maximum commercial content of glycerin should be carried therein. And if soap in any considerable amount is made from stock of good quality by this process, the highest efficiency of manufacture requires that the glycerin be separated. The necessity of good stock for best results, unless the product is to be further treated, and the value of glycerin have been chiefly instrumental in restricting the use of the process. Considered in its possibilities, the process has a broader field of application than the cold process and may be employed in the manufacture of soft soap either for domestic or industrial purposes, for the base of cheap milled toilet soap, for toilet-soap as the so-called castile, olive-oil and olive-oil foots soap, for marine or cold-water soap, for the base of soap powder and scouring-soap, and for a great variety of soaps which derive their popular names from the nature of the material added to them.

For the preparation of small batches a steam-jacketed kettle or crutcher may be used, but for large amounts, as may be required for soap powder and scouring-soap, a steam-heated kettle, such as is used for regular soap boiling, is necessary.

Since the development of practicable processes for the recovery of glycerin the semi-boiled process has become almost obsolete and survives only in modified forms of the cold process already described. Its practical use on a large scale is confined to the manufacture of soap for soap-powder and scouring-soap, although in many plants manufacturing exclusively cheap toilet-soap of the milled variety, and where the recovery of glycerin is no desideratum, it still persists. Where stock of any quantity is killed for soap-powder it is generally grained and the stock lye withdrawn. The same should be true of stock for scouring-soap. It is claimed that a more compact and less friable cake can be produced from a semi-boiled soap, and the process persists in certain plants in spite of the superior economy of separating glycerin. Were it not for the greater tendency of the soap to "sweat," owing to the affinity for moisture of the retained glycerin, it would be impossible to distinguish well-made milled toilet-soap prepared from a semi-boiled base from similar soap prepared from a base made by the grained process. The manufacture of scouring-soap from a semi-boiled base will comprise the method of manufacture described under this head, the methods of manufacturing other soaps being described under the processes by which they are at present commonly made.

Scouring or Sand Soap.—There has developed a large demand for a soap for scouring and polishing purposes which is supplied by a simple mechanical mixture of soap, silex, and soda-ash. The soap forms the binding agent, the silex the abrasive agent and the combined soap and soda-ash effect the chemical cleansing as distinguished from the mechanical.

The silex, or silica, referred to* is the product of rock-crystal quartz; the quartz is first to be brought to a white heat in a calcining kiln, then immersed in water, crushed under chasers, ground to an impalpable powder, and floated into settling-vats; from these it is to be dug out and dried until it reaches the condition known as "bone dry," when it is to be passed through the finest quality of silk bolting-cloth. The impalpable powder must present under the microscope the appearance of sharp annular needle-pointed particles; it must be non-absorbent and must not have been produced from infusorial or silicious earth.

The stock may be either cocoanut-oil or a mixture of oil and tallow, although any considerable substitution of the cocoanut-oil by tallow, is at a sacrifice of compactness of the finished product. The soap may be made by either the boiled or semi-boiled process; the elimination of glycerin from the stock does not effect in the least the character of the product. The procedure of manufacture by the boiled process will be described in the section devoted to grained soaps.

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* Navy Department specifications for sand soap.

Selection of Stock for Scouring-soap Base.—The selection of stock for the soap-base of scouring or sand soap is determined by the properties desired in the finished product. The essential requirements of a good scouring-soap are firmness and compactness, quick-lathering properties, homogeneity, dryness, high durability in use whereby the bar wears to the thinness of a wafer, and the uniform and fine state of division of the abrasive agent mixed with the soap.

Tallow forms a soap which when mixed with the abradent forms a firm mass of high durability but of low lathering power and inferior compactness, whereby the bar on becoming hydrated in use becomes brittle if not already so in the dry, unused bar.

Cocoanut-oil forms a firm and compact mass with the abradent, which possesses quick-lathering properties, but owing to the ready solubility of the soap, the bar tends to waste away more rapidly in use. Cocoanut-oil alone, or with a small proportion of tallow not exceeding 25 per cent, forms the most satisfactory base of a scouring-soap, the natural firmness of the soap and its capacity of absorbing large quantities of salts without separation constituting the advantage. As cocoanut-oil requires for saponification 17.5 to 19.0 per cent of caustic soda, by reference to the tables on pages 17 and 97 it will be seen that equal parts of oil and caustic-soda lye of about 25° Bé. density, prepared from 74° Bé. caustic, will be required for complete saturation. Similar combinations may be worked out for tallow if used in admixture with cocoanut oil. All scouring-soaps contain saturated soda-ash solution prepared as already described for the filling of grained soap. It hardens the soap, aids homogeneity of mixture, adds to the detergency, and makes the soap more applicable for use in hard and salt waters, and generally more efficient for scouring purposes. The proportion added will be determined by the degree of efflorescence of the finished soap on the market. Sodium silicate in small amount is a desirable addition. It imparts firmness to the soap, retards efflorescence, makes a more compact bar on drying, and adds some to the detergency. The proportions of these ingredients to be added depends entirely upon the stock and the appearance and consistency desired in the finished product.

In the manufacture of semi-boiled soap for use as a soap base for scouring-soap, of which cocoanut-oil is a part or exclusive stock ingredient, to which an alkaline salt filling is added, the addition of the latter is most advantageously effected, either in part or whole, in the soap-kettle, for thereby the saponification of cocoanut-oil is made much easier, the chief care then being to adapt the charge to the capacity of the kettle, so that the swelling which takes place on saponification will not cause the contents of the kettle to boil over. With such a procedure the saponification of cocoanut-oil or mixed stock becomes a comparatively simple operation, there being required only the simple addition of the ingredients in regulated amount, their mixing during the addition, and close watch to avoid boiling over.

The Abrasive Agent.—Ground quartz, or silex, is the most satisfactory abrasive agent for use in scouring-soap. Diatomaceous earth and marble-dust may be used. Ground quartz, or silex, is simply silica, SiO_2, an inert body of no detergent power in itself, but when reduced to a fine state of division acts as a mechanical abradent. When used for scouring-soap the quartz should be ground to an almost impalpable powder and with particles of uniform size and free from lumps, whereby dryness is required, so that the object to be cleansed will not be scratched, which property is especially desirable in a soap used for cleansing and polishing metallic objects. The amount of silex that can be incorporated with soap depends upon the stock, the amount of filling, and the firmness and compactness desired in the finished product and is upwards of 200 per cent.

Killing Stock.—In a scouring-soap, the soap is simply the vehicle for the abrasive agent. After the preparation of the soap-base there are two general methods of manufacture, viz., the frame and the mould methods, the former being used for the manufacture of the cheaper grades.

By the semi-boiled process, the following formula may be used for the soap-base with good results:

Cocoanut-oil, Ceylon, or copra..........	100 pounds
Caustic-soda lye, 25° Bé................	100 "
Soda-ash solution, 36° Bé...............	100 "

For small batches, saponification may be effected in a steam-jacketed crutcher or kettle. For large batches, a tank or kettle provided with open steam-pipes and suitable connections suffices. The requisite amount of oil, lye, and soda-ash solution having been prepared, the oil and lye may be added and heat applied. They may be added either simultaneously or successively, or a portion of

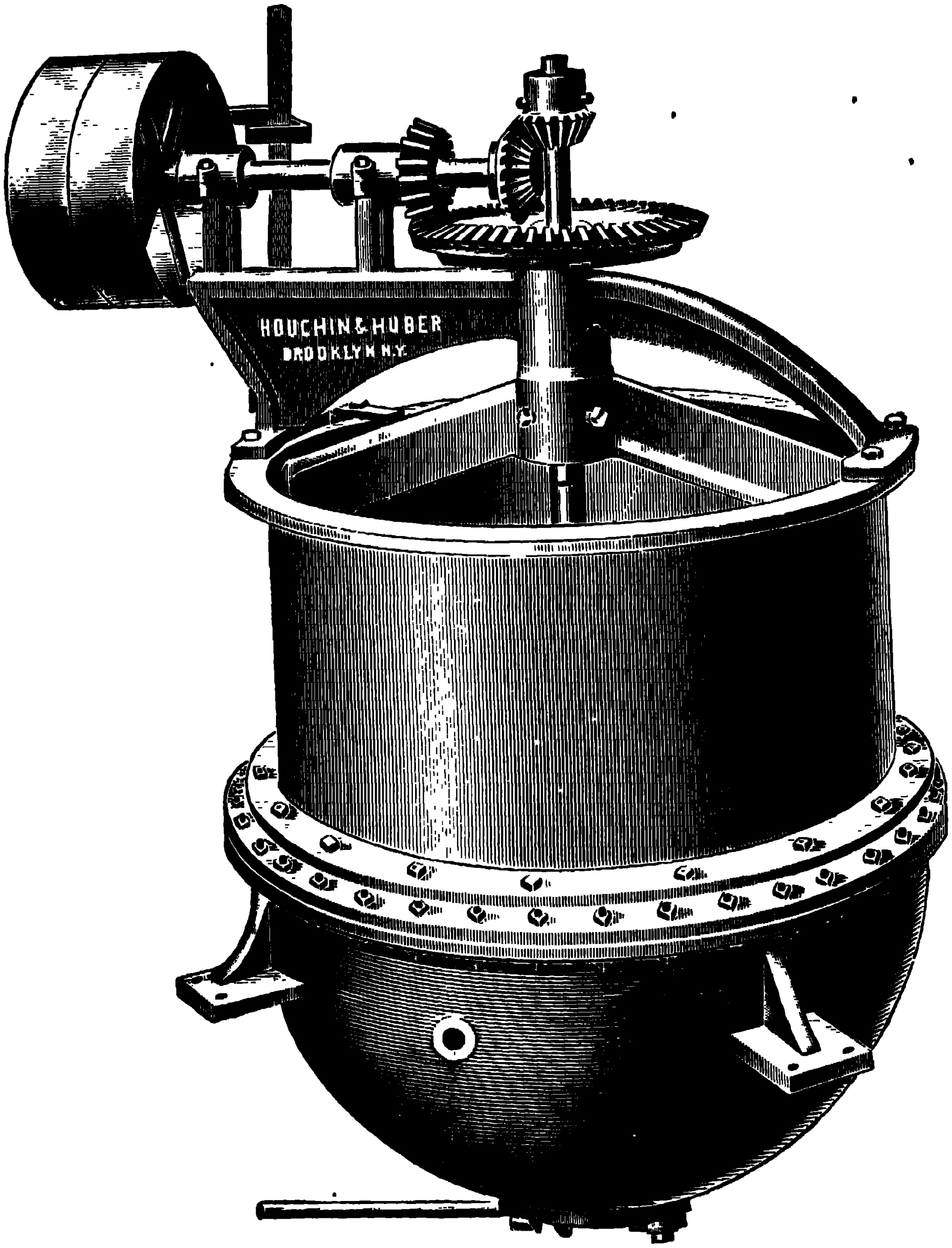

FIG. 130.—Scouring-soap Mixer.

the lye may be reserved to correct any tendency of the soap to boil over as a result of swelling incident to saponification. The alkaline filling may be also used for this purpose. When danger of boiling over has passed, boiling should be continued until all the strength is absorbed. More caustic should be added, but in no greater excess than that indicated by a slight taste. At the end the soda-ash solution may be added and well mixed through, if not added previously.

Mixing.—With sand soap made in small amounts, the silex may be added in the mixer in which the soap is made. This requires a strong, well-made machine, as the mixture is heavy. Otherwise the soap is pumped hot to a specially constructed mixer provided with a steam-jacketed bottom, Fig. 130, agitation being effected by a vertical shaft in which horizontal arms are spirally inserted. A shallow form of mixer is shown in Fig. 133, but this is more commonly used for preserving the mixed soap at a proper temperature prior to transferring it to the moulds. In a mixer of the type shown in Fig. 130 silex is added and thoroughly incorporated, any tendency to stiffen being corrected by the addition of soda-ash solution. To the proportions given for the filled soap the following proportions are added in the mixer:

Silex	600	pounds
Soda-ash solution, 30° Bé.	40	"

When the mixture is smooth, homogeneous, and of the proper consistency it is ready to be discharged from the mixer. Scrap may be worked over as fast as it accumulates by adding it to fresh batches in the mixer. Additional water or soda-ash solution must be added to correct dryness. From this stage two methods of scouring-soap manufacture originate, viz., the frame method and the mould method.

Frame Method.—From the mixer the mass is discharged into specially constructed frames or boxes in which the soap cools very quickly. As soon as the soap has acquired the desired firmness it is stripped and slabbed. A slabber for this purpose is shown in Fig. 131. A cutting-table especially constructed for use with scouring or sand soap is shown in Fig. 132. In front of the second cutting-head is a printing-roll, or cylinder, with plates attached which stamps the soap as it passes through the second cutting-head. A cutting-table of this type will cut and stamp cakes of scouring-soap at the rate of three to five slabs per minute, or as rapidly as the cakes can be removed from the table.

Mould Method.—The hot mixture of soap, silex, and soda-ash solution is discharged from the mixer and descends by gravity to the mould-room, where it is collected in a shallow steam-jacketed kettle, Fig. 133, and kept in a viscous or soft state until used. The mixture is transferred to moulds, Fig. 134, over which it is carefully smoothed so that each compartment is filled. If the soap cools too much and becomes stiff it cannot be satisfactorily moulded. The moulds, Fig. 134, resting on thin sheet-iron slabs, are transferred to racks to await the solidification of the soap. When firm enough the mould and slab are inserted beneath the multiple dies of the press, Fig. 135; the mould is supported by a flange while the slab rests upon the base-plate of the press, which corresponds to the bottom die. Pressure is exerted simultaneously by the attendant by means of the foot below and the screw above. Pressure is first released from below, whereupon the

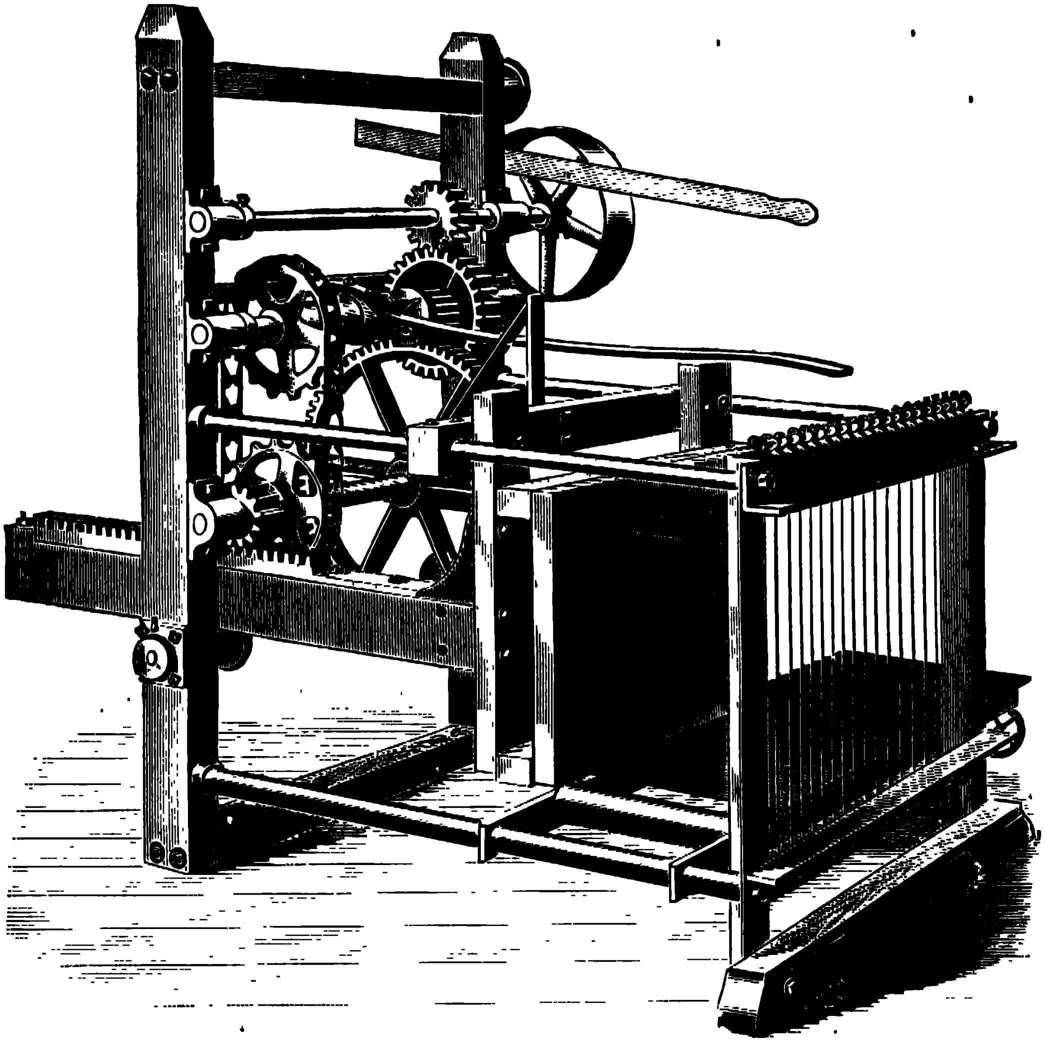
FIG. 131.—Power Slabber for Scouring-soap.

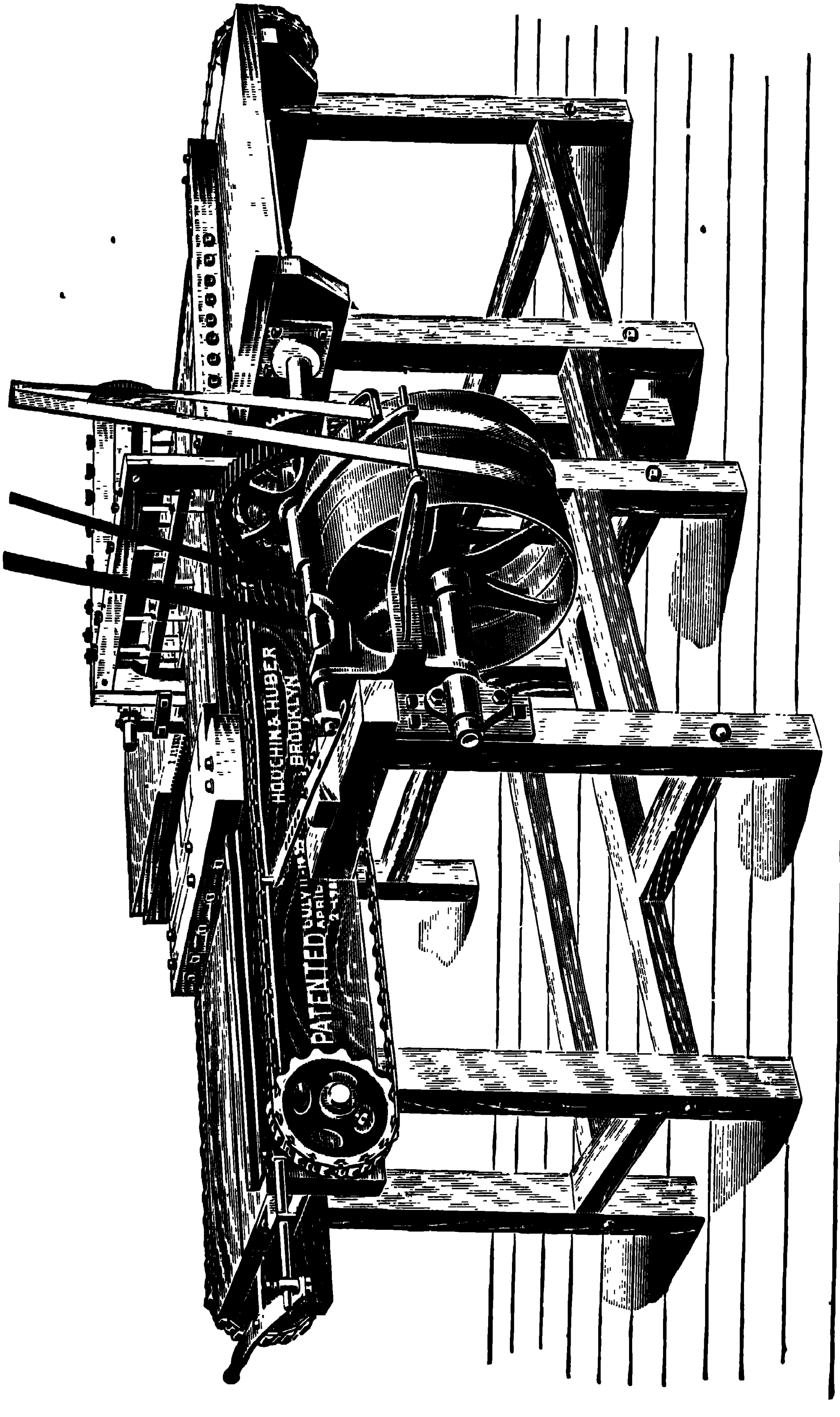

FIG. 132.—Cutting-table Designed for Scouring-soap

pressed cakes drop from the mould onto the slab below. The cakes resting upon the slab are stacked in piles on a truck and transferred to the drying-room, while the moulds as rapidly as emptied are hauled to the fillers for use again.

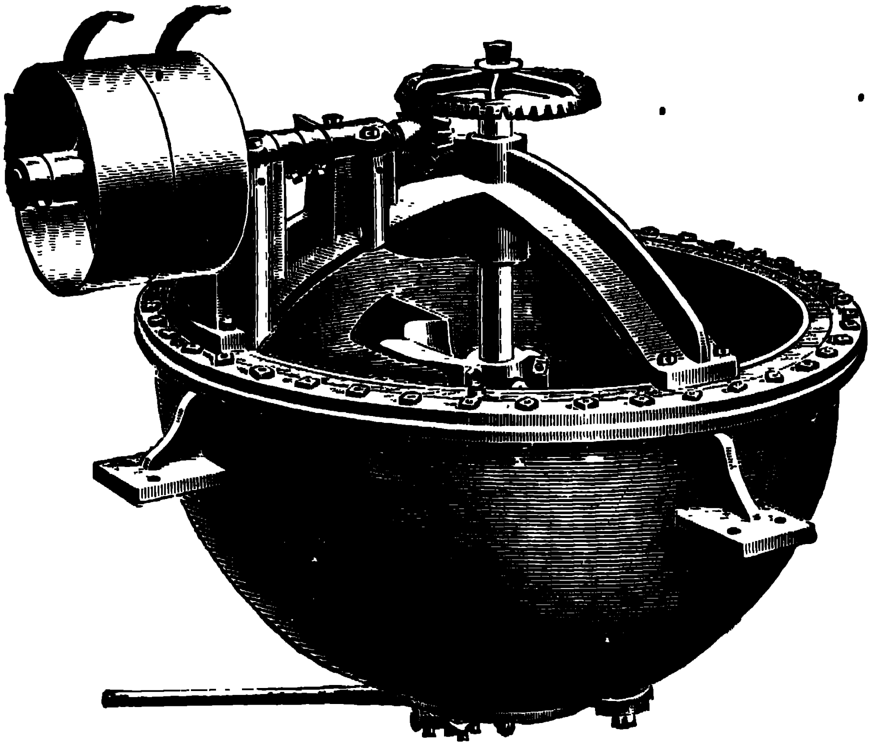

FIG. 133.—Mixing- or Heating-machine Jacketed, with Large Opening in the Bottom.

Scouring-soap Press.—In the operation of the scouring-soap press, Fig. 135, the mixed material is spread into the divisions of the mould, shown in Fig. 134, supported on a metallic plate which serves as the bottom. The material should be of a consistency that will permit of ready flowing, so that each compartment of the mould may be filled without the occlusion of air, which if retained will form a cavity in the cooled soap and thus form an imperfect bar. When each compartment has been filled and the material spread in a thin layer over the surface the filled mould is placed on racks and allowed to remain until the soap is sufficiently firm to slip readily from it and to receive the impression of the die. Previous to stamping, the surplus soap is cut from the surface with a long-bladed, two-handled knife and the mould then inserted in the press (Fig. 135).

Simultaneously with the insertion of the mould, the bedplate of the press is elevated by means of pressure applied by the foot of the operator on the lever below and the multiple die lowered by the rapid motion of the screw, whereby the distinguishing characters are impressed in the surface of each bar. By removing the pressure from the lever, the removable bottom of the mould falls upon the bedplate of the press and the cakes, as a result of the pressure yet applied from above, are forced from each compartment and fall upon the separated bottom. The screw is then elevated and the plate of pressed soap is transferred to a truck, which when filled is removed to the drying-room. The mould is now removed and is ready for use again with an extra bottom. After the lowering of the bedplate of the press, as described, the mould remains stationary, supported by the rim, as shown on the opposite ends.

Drying Scouring-soap.—Scouring-soap should be bone dry before wrapping, otherwise it will be friable and disintegrate rapidly in use. It is desired that each cake wear thin. To effect this more prolonged drying, and at a higher temperature than is permissible with ordinary soap, is required. For this purpose drying-rooms of various types are erected, but the principles of drying are the same as for ordinary soap. These are discussed elsewhere.

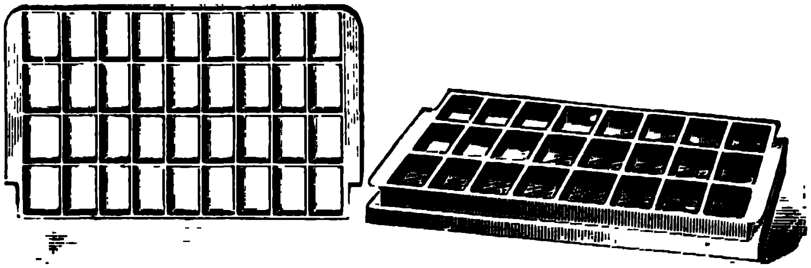

FIG. 134.—36- and 24-cake Mould with Slab.

The drying period, however, is longer, for the soap is rendered practically anhydrous, its durability in use being thereby increased. The scrap comprises scrapings from the moulds, imperfect bars, and returned soap. It is remelted in the ordinary type of remelter, more heat being required, however, owing to the greater firmness of the soap, and returned as fast as remelted to the crutcher, where it is incorporated in the regular mixings.

Soft Soap.—Soft soap may be defined as soap made from stock of low melting-point by use of caustic potash. Such soap, according to the temperature and nature of stock used, is of varied color, more or less transparent, soft, unctuous, of faint soap-like odor and of ready solubility. The manufacture of soft soap for domestic or industrial purposes in the United States is very limited; in England and European countries its manufacture constitutes a considerable business. As a manufactured detergent it has been almost entirely displaced by soap-powder. Modern practice has compelled the distinction that while all potash soaps are soft soaps, all soft soaps are not exclusively made with potash.

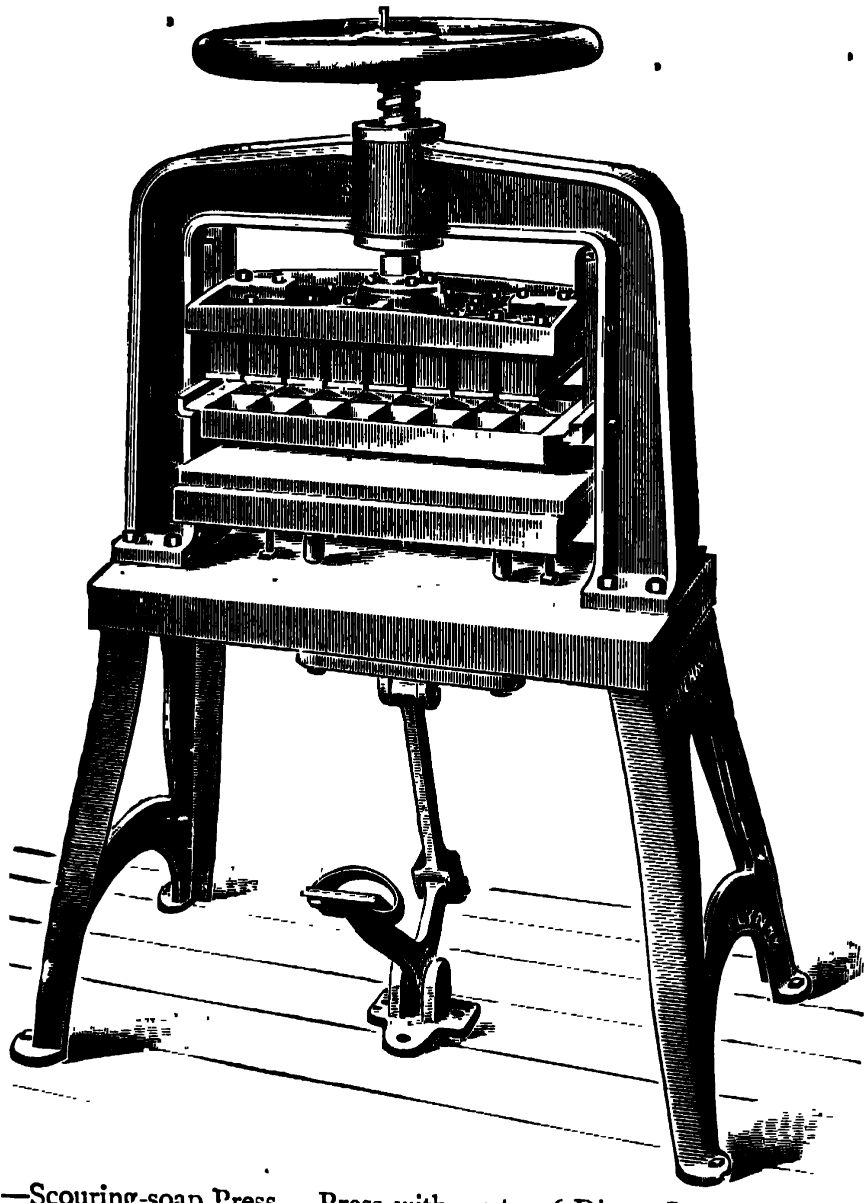

FIG. 135.—Scouring-soap Press. Press with 24 to 36 Dies to Impress and Eject the Cakes from the Compartment Moulds.

Stock.—The nature of the stock used is determined by the purpose for which the soap is intended, linseed-, cottonseed-, and corn-oils, olive-oil, olive-oil foots, and tallow being commonly used. In the preparation of soft soap a potash-linseed-oil soap is the only combination that will retain its transparency throughout summer and winter. Owing to the greater cost of linseed-oil, cottonseed-oil, alone or in admixture with tallow in various proportions, generally is used. A potash soap of these ingredients will be transparent, with a light-golden color in warm weather and at lower temperatures will show a "figged" appearance from the crystallization of potassium stearate and palmitate in the menstruum of potassium oleate. Unless completely saponified, soft soap soon becomes rancid with the development of odor characteristic of the stock used. Linseed-oil saponifies readily, forms a pale-colored, clear soap of soft body, keeping well, but with the odor characteristic of the oil used. It is the basis of all soft soaps in regions where these soaps are largely used, the proportion depending upon the season and the price of competing olein-stock. Palm-oil is little used as a stock ingredient, serving mostly as a coloring agent and perfume, either killed with the stock or added after saponification.

Olive oil is an excellent ingredient for soft soap, but owing to its high cost it is seldom solely used except for soap intended for pharmaceutical purposes. Rosin may be used in small proportions in combination with stearin-containing stock, the solubility of the soap, hence its lathering properties and detergency, being thereby increased. It serves as a coloring agent, and when used in small amount with linseed-oil tends to modify the odor of that oil. Red oil of good quality forms an odorless soap. It possesses an advantage in that it may be killed with carbonate of potash. Tallow, as a rule, is not used alone, but always in combination with olein-stock. Cocoanut-oil forms an almost colorless, transparent soap, but unless completely saponified rancidity soon develops, which, however, will occur eventually under any condition. For clear, transparent soap, very soft stock and caustic potash are essential. Figging may be effected by either introducing a portion of stearin-containing stock or by substituting caustic soda for a portion of the caustic potash. By the use of tallow alone a white potash soap may be made. The percentage absorption of caustic potash by the various fats and oils is 1.4 times greater than that of caustic soda.

Procedure.—A good soft-soap figging well in summer and fairly soft in winter, with the desired soft texture when pressed between the fingers or withdrawn from a mass of the soap, may be made from two parts of cottonseed-oil and one part of tallow or three parts of cottonseed-oil and one part of tallow, the latter combination forming soap of softer consistency. Saponification with caustic potash proceeds as on the stock charge of all boiled soaps. The oil and tallow are run in together, and boiling having started, caustic-potash lye at 10°-15° Bé. is added. When saponification has begun, as indicated by the formation of an emulsion and the mass becoming clear, lye is added continually, but never in sufficient excess to grain the soap as long as it is absorbed. With strength present in permanent excess, boiling is continued until satisfied of complete saponification. Where it is possible to measure with some degree of accuracy the proportions of ingredients required, the manufacture of semi-boiled soap involves no great difficulty, it being sufficient to run the ingredients into the kettle according to the procedure stated and boil until combination is effected. It is then known that the excess or deficiency of alkali is very slight. But under ordinary conditions the amount of stock to be killed is the only factor known with accuracy, the skill and experience of the soap-maker being relied upon entirely to manipulate the ingredients and to determine the completion of saponification, with neither free fat nor free alkali in excess. After the completion of saponification, to obtain a product of the desired consistency and yield, the addition of water to the soap in the kettle should not be in such excess that on cooling a sample the short texture is destroyed. The presence of water in excess of the proportions desired will be indicated by the cooled sample pulling apart in a stringy, thread-like mass. When finished the soap is pumped to the crutcher, where it is agitated merely to cool it, whence it is run directly into barrels, kegs, or other packages stored in a cool place.

A cheaper soft soap for industrial purposes may be made from the same ingredients, or from cottonseed-oil alone, by substituting caustic soda for a portion of caustic potash. The degree of substitution will be determined by the consistency desired in the finished product. The finished soap is pumped directly to the crutcher, where it is strengthened by the addition of a small amount of 20°

Bé. caustic-soda lye or a mixture or caustic-soda lye and soda-ash solution of 36° Bé. While the addition of these compounds dilutes the soap with water to any desired consistency, the alkali imparts a body to the soap that it would not otherwise possess, and for many purposes a sharp strength is desired. In no case should it be added in amount sufficient to grain the soap.

When rosin is used it is added and killed after the saponification of the glyceride stock. It combines readily with alkali and serves well to take up excess strength.

Yield.—The yield of soft soap of a definite consistency from a given stock decreases in proportion as caustic soda is substituted for caustic potash, not alone because less caustic soda than caustic potash is absorbed, but because hydration of the resulting soap is reduced. The inferior affinity of soda soap to potash soap for water affects the yield of soft soap when the former forms a part of it. Linseed-oil and caustic potash will yield 240 pounds of soap to 100 pounds of stock, but with one-third of the lye caustic soda, the yield is reduced 10 to 15 pounds. The yield is greater with linseed-oil than with cottonseed oil, and is greater with cottonseed-oil alone than with tallow in admixture; the firmer the glyceride the smaller the yield of soap of the texture desired.

Soft soap is commonly considered to yield from two and one-fourth to two and one-half times the weight of stocks used, but it may be thinned out with water to any desired consistency. The yield may be increased by the addition of filling material. As high as 30 pounds of potassium-chloride solution can be incorporated. A yield in excess of 270 pounds can be obtained only by the use of binding agents as starch solution. With unfilled soft soap the proper degree of hydration and yield are obtained, when a soap of short texture is produced as previously described. The presence of water in excess of the yield stated will destroy the short texture and make the soap stringy. The alkaline salts used for filling comprise the chlorides, carbonates, and silicates of soda and potash. The potash salts for this purpose are preferable owing to their affinity for water.

CHAPTER VIII.

GRAINED SOAP.

Classification. Grained Process. Graining Agents. Class I. Manufacture of Soap-powder Base: Stock Change. Graining. Strengthening and Finishing. Mixing and Framing. Grinding Soap-powder. Packing Powder. Continuous Method. Scouring-soap Base: Stock Change. Graining. Strengthening and Finishing. Classes II and III. Influence of Detergents on Textile Fibres. Cotton. Wool. Textile Soap. Wool Textile Soaps. Scouring Raw Wool. Scouring the Woollen Fabric. Milling the Woollen Fabric. Silk Textile Soap. Cotton Textile Soap. Stock for Textile Soap. Soap for Printers' and Dyers' Use. Manufacture of Textile Soap. Red Oil. Combination with Caustic Soda. Combination with Soda-ash. Castile Soaps. Marine or Salt-water Soap. Boiled-down Soap. Procedure. The Mottle. Mottled Soap. Mottling Agents. Filled Mottled Soap.

Classification.—As noted on preceding pages soap-manufacturing processes have been divided into two general and distinct classes, viz., the cold and boiled processes. The boiled process has again been subdivided into the semi-boiled and grained processes. Particular stress is laid upon this classification because it is desired that some system and clearness of understanding be introduced into the phraseology of the processes of soap-manufacture. By some a semi-boiled soap is considered one that has been made at a slightly higher temperature than that prevailing in the manufacture of cold-process soap. While there is much similarity between the cold process and that procedure that is carried out at a slightly higher temperature, chiefly in that the mechanical equipment required for both is the same and that both methods of treatment are adapted only for small amounts of material and that on a small manufacturing scale, the old distinction prevailing to a greater or less degree has been disregarded in this work as unscientific and conducive to misunderstanding. The term "settled" is generally ascribed to a soap made usually by killing the stock, graining the soap, rosining it if it be a rosined soap, strengthening it, and finally leaving the contents of the kettle in such condition that, on cooling, the impurities "settle" to the bottom of the kettle. Such a distinction is equally confusing, for even though a soap be not grained, it can, if desired, be left in such a condition after saponification of the stock that practically the same purification is effected; and also a grained soap can be framed without settling.

By basing a secondary classification on the use of salt as a graining agent in the sense in which it was originally used before glycerin was thought of as a commercial by-product in the manufacture of soap, viz., to purify the soap, a classification that leaves no confusion is obtained. As already noted, the same term is applied to the soap as well as to the process by which the soap is made. Therefore by the boiled process saponification is induced and effected by boiling the ingredients by means of artificial heat. Soap made by the boiled process may be subdivided into two general classes, viz., semi-boiled and grained. A semi-boiled soap is one from which the glycerin has not been separated and the process by which it is made has come to be in modern practice of very limited application. A grained soap is one from which the glycerin has been separated and the process by which such soap is made is the most important and general of all soap-manufacturing processes. A grained soap may or may not be a settled soap, as we shall see in the manufacture of mottled soap. The general application of practicable processes for the recovery of glycerin from waste soap lyes, wherever such can be economically treated, has revolutionized the art of soap manufacture.

In the following diagram has been arranged an outline of the four general processes of manufacturing grained soap, under the successive discussion of which will be described the procedure of manufacture of the various kinds of boiled grained soap.

TABLE XIX.—OUTLINE OF THE FOUR GENERAL CLASSES OF PROCESSES FOR THE MANUFACTURE OF GRAINED SOAP.

Class.	Materials.		Common Name of Change.	Product of Change.	By-product of Change.
	Used.	Used but Removed.			
I.	Stock and caustic lye	Salt	Stock	Grained soap	Stock lye
	Caustic lye		Strengthening and finishing	Soap	
II.	Stock and caustic lye	Salt	Stock	Grained soap	Stock lye
	Caustic lye	Caustic lye	Strengthening	Strengthened soap	Strength lye
	Water		Settling	Soap	Nigre
III.	Stock and caustic lye	. Salt	Stock	Grained soap	Stock lye
	Caustic lye	Caustic lye	Strengthening	Strengthened soap	Strength lye
	Brine	Brine and caustic lye	Pickle	Neutral soap	Pickle lye
	Water		Settling	Soap	Nigre
IV.	Stock and caustic lye	Salt	Stock	Grained soap	Stock lye
	Rosin, caustic lye, and salt	Salt and caustic lye (?)	Rosin	Rosined soap	Rosin lye
	Caustic lye and brine	Caustic lye and brine	Strengthening	Strengthened soap	Strength lye
	Water		Settling	Soap	Nigre

Grained Process.—It will be observed that the subject has been developed logically along the line of simplicity of manufacture; that a greater variety of stock and of different grades becomes available, until with the grained process we reach the maximum in this respect. While under all circumstances superior stock is necessary for the best results, the grained process is essentially one to be used for inferior stock. The discussion of soap making by this process will be from two points of view, (1) the elimination of impurities from the soap, and (2) the separation of glycerin from the stock and soap. In the cold and semi-boiled processes glycerin remains as an ingredient of the soap, and where other signs are uncertain the analytical determination of the quantity present is a reliable index of the origin of the soap; while in the grained process every effort is made to effect the maximum separation of glycerin and its completest recovery from the waste lye formed by graining. In the discussion of the grained process the manufacture of the various grades of soap made by it will be described in the order of their relative simplicity of manufacture. And it will be observed that the skill of manipulation and complexity of procedure requisite to a product of given quality are in inverse proportion to the quality of stock used.

Graining Agents.—Graining soap is throwing it out of solution, the degree of separation depending upon the proportion of the precipitant present. For the production of a definite curd or grain common salt combines most fully cheapness and efficiency. Caustic soda is used as a graining agent only on the strengthening change, and then only incidentally, the lye from which is used again, whereby the caustic is recovered. It is a more efficient graining agent than common salt, meaning thereby that a smaller amount is required to produce the same grain. Sodium sulphate, or Glauber's salt, produced in the soap industry by the use of sulphuric acid in the neutralization of the alkalinity of waste lye preparatory to evaporation, is inferior to common salt. In a number of experiments made to determine the relative efficiency of common salt and Glauber's salt, 5000 grams of settled rosined soap of good quality were grained with both agents to an identical curd, whereby it was ascertained that equal quantities of soap used required 150 grams of salt and 250 grams of Glauber's salt to produce the same grain. Therefore, according to this experiment, two-thirds more Glauber's salt than common salt are required to grain soap. From these experiments it is calculated, on the basis of the weight of closed soap, that 3 per cent of common salt and 5 per cent of Glauber's salt are required for graining. These proportions, while approximating, are less than those prevailing in actual practice. Glauber's salt is not used as a graining agent only in so far as it may occur as an impurity in salt recovered from waste lye the alkalinity of which has been neutralized with sulphuric acid.

Soap-makers are accustomed to speak of a "thick" or "thin" curd, of an "open" or "close" curd, or of a "short" or "broad" curd, meaning respectively the two extremes of separation of soap and lye according to the amount of salt present. Salt may be used either in the dry state or as a saturated solution, viz., 25° Bé. The percentage of dry salt in salt solutions of different densities at 60° Fahr. is shown in the following table.

TABLE XX.—PERCENTAGE OF DRY SALT IN SALT SOLUTIONS OF DIFFERENT DENSITIES AT 60° FAHR.

Degrees Baumé.	Specific Gravity	Per Cent of Salt.	Weight of a Gallon of this Brine in Pounds.	Pounds of Salt in a Gallon of Brine of 231 Cubic Inches.
.26	1.002	.265	8.347	.022
.52	1.003	.530	8.356	.044
1.04	1.007	1.060	8.389	.088
1.56	1.010	1.590	8.414	.133
2.08	1.014	2.120	8.447	.179
2.60	1.017	2.650	8.472	.224
3.12	1.021	3.180	8.506	.270
3.64	1.025	3.710	8.539	.316
4.16	1.028	4.240	8.564	.364
4.68	1.032	4.770	8.597	.410
5.20	1.035	5.300	8.622	.457
7.80	1.054	7.950	8.781	.698
10.40	1.073	10.600	8.939	.947
13.00	1.093	13.250	9.105	1.206
15.60	1.114	15.900	9.280	1.475
18.20	1.136	18.550	9.464	1.755
20.80	1.158	21.200	9.647	2.045
23.40	1.182	23.850	9.847	2.348
26.00	1.205	26.500	10.039	2.660

It is used in a dry state on the stock and rosin changes and in solution on the strengthening change. When used to wash the soap for the removal of free alkali or coloring-matter, whether it is in solution or in a dry state, will depend upon conditions in the kettle. "Thick," "open," or "short" grain is produced by an excess of salt; with a deficient amount the texture is described as "thin," "close," or "broad." The amount of water withdrawn from the soap, likewise the amount of waste lye withdrawn from the kettle, is determined by the quantity of salt used in graining. In good practice it is endeavored to secure the best separation of the maximum volume of waste lye with the least amount of salt. The degree of separation is determined largely by the quality of the stock and the nature of the change. With inferior stock highly charged with albuminous matter as thorough a separation as is practicable is desired, whereby the utmost impurity may be discharged into the waste lye. The same conditions prevail in graining the nigre; hence a thick, open, or short curd should be produced under such conditions. The use of inferior stock not only means less glycerin actually present, and, other things being equal, more salt required for graining, but as well a larger volume of waste lye to be evaporated and salt to be recovered for the smaller yield of glycerin. The economy of good stock is evident. From 6 to 8 per cent of dry salt, on the basis of the stock used, will produce a waste lye of about 13° Bé., which with good practice will contain the maximum amount of glycerin with a minimum of salt, leaving the soap sufficiently grained. The use of caustic potash as a saponifying agent requires that the soap be made by either the cold or semi-boiled process, for the use of common salt as a graining agent for such soap involves a loss of a portion of the potash by the interaction of the potash soap and sodium chloride. The use of potassium chloride as a graining agent is impracticable. It has been demonstrated* that under the conditions mentioned the reaction occurs according to a general rule, viz., that when potash and soda are simultaneously present in contact with two acids jointly equivalent to the sum of the two alkalies, there is a marked tendency for the potash to unite with the stronger acid and for the soda to unite with the weaker one. Hydrochloric acid being a stronger acid than the ordinary fatty acids, it results that if fatty acids and hydrochloric acid constitute the pair, the potash unites by preference with the hydrochloric acid, and the soda with the fatty acids, forming soda soap.

It is to be noticed, in connection with this general rule, that we find that under certain conditions changes may be brought about which are apparently in opposition to it; in point of fact the two acids and the two bases always so associate themselves as to give rise to *four* salts, viz., those obtainable by the combination of each acid with each base; and by varying the relative masses of the acids and bases in certain ways, one pair of salts can be made to predominate over the complementary pair under one set of conditions, and *vice versa* under other conditions. For example, we find that if a soda soap be dissolved in water and a large quantity of potassium chloride added to the solution, it is possible to convert considerably more than half of the soda soap into potash soap, with formation of the corresponding quantity of sodium chloride, out of the four compounds, (1) potash soap, (2) soda soap, (3) potassium chloride, and (4) sodium chloride. Under these conditions 1 and 4 are formed to a larger extent than 2 and 3, so far as the fatty acids and soda are concerned; while if a potash soap be similarly treated with sodium chloride the same four salts will result, only now 2 and 3 will be formed to a greater extent than 1 and 4, so far as the fatty acids and potash are concerned, the difference in the two cases being essentially due to the fact that, in the first instance, a relatively large mass in excess of 3 is present and in the second a similar excess of 4.

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* Wright and Thompson, Jour. Soc. Chem. Ind.

In a similar way, if a potash soap be well intermixed in a fluid condition with a solution of carbonate of soda, and if, in a parallel experiment, a soda soap be intermixed with carbonate of potash, in each case four compounds will result, viz., (1) potash soap, (2) soda soap, (3) carbonate of potash, and (4) carbonate of soda, the relative proportions in which the two alkalies are associated with fatty acids depending on the mass and nature of the alkaline carbonate present. In this instance, however, the displacement of potash from combination with fatty acids by soda always takes place less readily, and that of soda from combination with fatty acids by potash more readily, than is the case when alkaline chlorides are used as above instead of carbonates.

It is remarkable that when there are two alkalis present (potash and soda) in the caustic state (i.e., as hydroxides), and no acid except the fatty acids of soap, there is no marked preferential combination of either alkali with the fatty acids as compared with the other. Thus we find that all the ordinary fatty acids, when treated with a mixture of caustic potash and caustic soda in equivalent proportions, and in quantity jointly equal to twice the amount capable of combining with the fatty acids, appear (according to our experiments) to form a mixture of potash and soda soaps, containing sensibly half the fatty acid in combination with the one alkali and half with the other, while the uncombined alkaline lye similarly contains equivalent quantities of each alkali.

Class I.—The simplest treatment of a grained soap involves merely the saponification of the stock, separation of the waste lye, and the closing of the soap preparatory to its further utilization as may be desired in the manufacture of soap-powder and scouring-soap base. It is preferable, however, to subject the soap to a strengthening change, which, with the soaps commonly made by this process, may be combined with a finishing change. Complete saponification is thus assured. As soap-powder base is made by this process attention will be first directed to it.

Manufacture of Soap-powder.—The demand for a cheap, quick detergent for the cleansing of coarse articles is met by a combination of soap, soda-ash, and sodium silicate with varying proportions of non-detergent matter. The success of early manufacturers of this article and its ease and cheapness of preparation encouraged a host of imitators until at present its manufacture is general and the use of the article steadily increasing.

Stock.—Any saponifiable stock is suitable for soap-powder. Soft-bodied stock as dark grease is commonly used, there being no limit to inferiority provided the stock is reasonably free from non-fatty matter and offensive odor. The character of the resulting soap, aside from the odor, is effectually concealed by the soda-ash. Cottonseed-oil soap-stock is exclusively used in a popular proprietary powder. Off cottonseed-oil forms an excellent stock either alone or in admixture with grease. Rosin under no circumstances is permissible, as owing to the deliquescence of rosin soap, the soap-powder will become lumpy. It is practicable, however, to utilize to a certain degree the nigre, first sharply grained and then closed, from a high-grade, lightly rosined soap. The extent to which this is permissible must be determined by results.

Stock Change.—Saponification of all animal stock, and vegetable stock with the exception of cocoanut and palm-kernel oils, should be started with weak lye not exceeding 15° Bé. With grease-stock, which is the least refractory towards alkali, all the stock intended to be used may be added at once and the lye at 15° Bé. afterwards, or they may be added simultaneously with vigorous boiling. An emulsion forms at once and saponification ensues with rapid absorption of alkali. The alkali should not be added in such excess as will grain the soap, for thereby, the soap being thrown out of solution, saponification will be retarded. The greater part of the lye should be added at 15° Bé. As the mass boils up evenly and clear, the strength of the lye should be increased to 18° Bé. until all greasiness disappears, when it should be added more cautiously, but always in slight excess, and as often as absorption makes it necessary. When the soap, pressed between the fingers, chips up smooth and dry with caustic taste, boiling is continued until causticity disappears. More lye in small amount at 18° Bé. is added with continued boiling until a slight caustic taste is permanent. The soap should be further boiled for an hour or more to again reduce the soap practically to a neutral state, when it is ready to grain.

Graining.—With the soap boiling well up in the kettle, dry salt is added with the shovel and scattered uniformly over the surface of the soap. After each addition of salt it should be well boiled through before subsequent additions are made, until a portion of the soap removed on a paddle separates lye. Boiling is now continued, without further addition of salt, until that already added has been thoroughly boiled through. Should the lye at this stage remain clear on cooling, sufficient salt has been added and steam may be turned off and the lye allowed to settle out.

In small batches of 5000 pounds, stock may be killed and grained before noon and the lye allowed to settle during the noon hour. With some strength in excess boiling may be continued slowly throughout the day and the soap grained late in the afternoon and allowed to settle overnight. After withdrawing the stock lye the soap is ready to strengthen and finish.

Strengthening and Finishing.—After withdrawing the stock lye, steam is turned on and weak lye of about 10° to 12° Bé. added. Boiling is continued without further addition of lye so long as the soap shows strength. Continued boiling with water of condensation from the steam may suffice to close the soap; if not water may be added to bring the soap to the proper degree of hydration. Soap for soap-powder should be finished thinner than is required for settling. When this condition has been reached the soap is ready to be pumped, or conveyed by trough from the discharge-pipe of the kettle, to the crutcher.

Strengthening may constitute a separate change if desired, in which event, after withdrawing the stock lye, steam is admitted and water carefully added until the soap closes. The soap is then grained with 18° to 20° Bé. lye and boiling continued for at least two hours. Steam is now turned off and the lye allowed to settle out. The strength lye is withdrawn to a storage-tank to be worked over. Steam is now turned on and water added until the soap is brought to the desired consistency.

Mixing and Framing.—For this purpose the soap-crutcher may be used, but it is desirable that for mixing the thick, heavy mass of soap and ash the mixer be more strongly built than the ordinary soap-crutcher and preferably of the type with either the horizontal or vertical blade agitator. The ingredients added to the mixer comprise thin soap, soda-ash, and silicate of soda. These may be in various proportions according to the quality of powder desired. For a single frame 600 to 700 pounds each of soap and ash may be used to which 100 to 125 pounds of silicate are added. Soap is run in first, the amount being determined by the level in the mixer, and then the ash in successive amounts until the entire weight is added. Addition of the entire amount of ash at once may block the crutcher and bend or break the blades of the mixer. The amount of silicate added will depend upon the con-

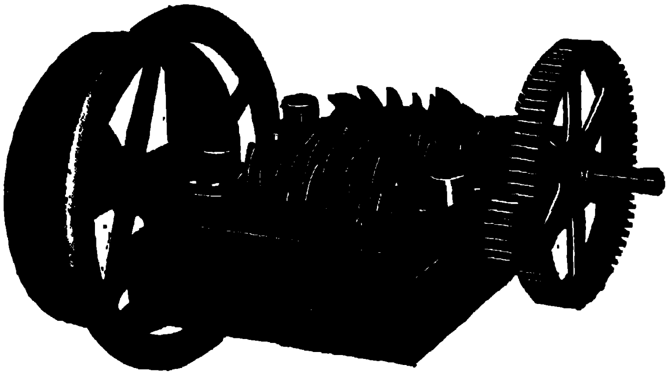


FIG. 136.—Disintegrator.

sistency of the mixture of soap and ash. If the soap is finished too thin, the weight of ash may not be sufficient to counteract the thinness of the resulting mixture, in which event either more ash or less silicate may be added. With soap finished coarse in the kettle it may be necessary to add water in the crutcher. When a homogeneous mixture has been obtained the mass is framed. One day, as a rule, suffices for cooling, at the end of which time the mass is stripped and cut. Corners and ends of the frame are usually hard, to penetrate which the ordinary soap-slabber is unsatisfactory. Recourse is had to hand-slabbing. Should the mass become too hard to be cut by wire, it must be disintegrated by more laborious means. After slabbing it is cut transversely and the slabs piled up to dry. The frame method is the most cleanly and convenient. According to the floor method, the mixed soap and soda-ash are run directly from the crutcher, used exclusively for this purpose, on to the floor of the apartment, where it is allowed to solidify, after which it is broken into coarse lumps to facilitate cooling and drying. The mass is then further disintegrated and ground as required. The slabs after disintegration, either by hand or disintegrator, are ready for grinding. A common type of disintegrator, or crusher, is shown in Figs. 136 and 137.

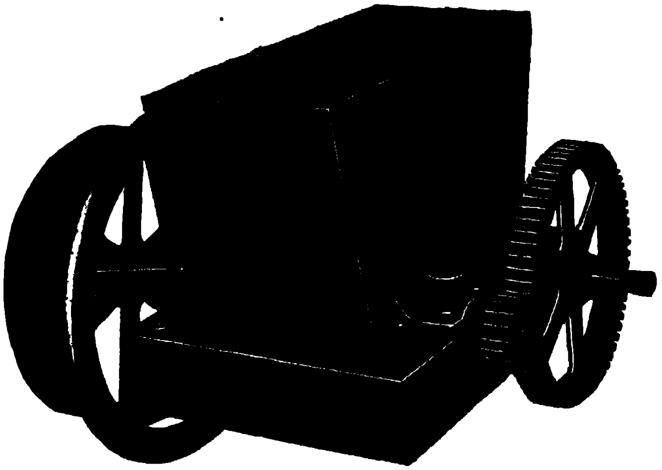


FIG. 137.—Disintegrator with Casing.

To avoid the troublesome process of framing the semi-liquid mixture of soda-ash, sodium silicate, and soap, allowing this mass to solidify in the frames and subsequently to reduce the solid material to a form suitable for grinding, it is possible to discharge the mass directly from the mixer to the grinding-mill. A device designed for this purpose is shown in Fig. 138. As shown it stands on a wooden frame which is securely stayed and bolted. It has two shafts running horizontally side by side, geared to run in opposite directions; they are run through a cast-iron case circling at the bottom, so that the paddles placed on the shaft in screw form lift the material from the bottom of the case and throw it in opposite directions from one end of the case to the other, thereby keeping it in constant motion and obtaining a perfect mixing.

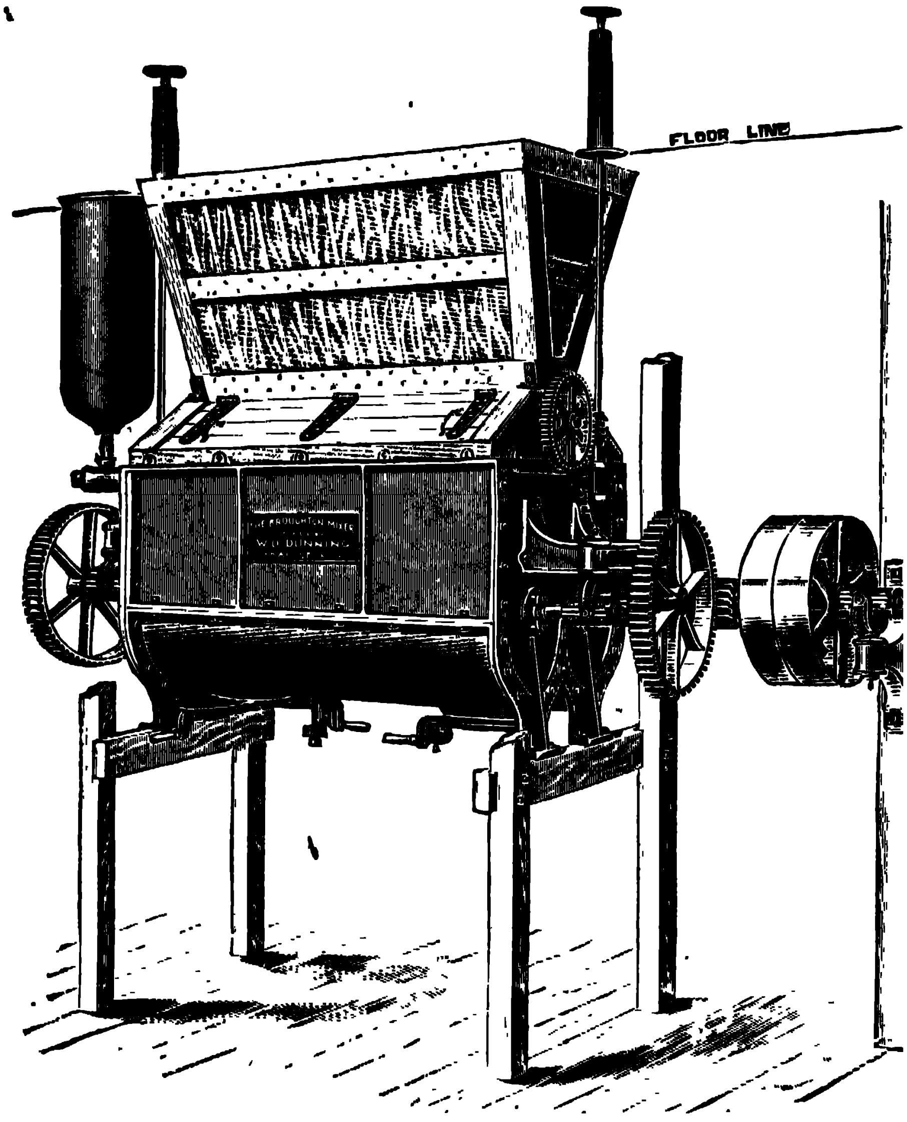


FIG. 138.—Soap-powder Mixer.

Where the shafts pass through, stuffing-boxes are provided to prevent any leakage. All bearings run in self-oiling boxes outside of case, so that the material does not come in contact with them, thus rendering them perfectly free from dust and durable.

The hopper on top of the mixer to receive the dry powder is provided with a door at the bottom, which is operated from above by a hand-wheel connected to the shaft, with a worm meshing into a worm-gear, to let the charge into the mixing-chamber. At the opposite end of mixer is a reservoir to hold a charge of hot soap. Attached to this is a pipe leading into the mixing-chamber; in this pipe is a quick-opening valve that is operated from above. Four tight-fitting gates are provided at the bottom of the mixing-chamber to discharge the material when mixed. This is done very rapidly. The consistency of the material as discharged from the mixer depends upon the proportions of solid and liquid ingredients added.

If the proportion is four to five parts of dry material to one of liquid, it will leave the mixer in a dry, powdered state and can be passed directly to the grinder. The soap should be heated to about 140° Fahr. If the proportion of the charge is about equal, the mixture will be in a partly dry state and will have to stand a short time to dry or to cool sufficiently for grinding.

As considerable heat is generated in the grinding process, it is best with material containing a large proportion of soap to allow it to thoroughly cool before grinding. Whether or not the material may be ground directly from the mixer is determined primarily by the proportion of soda-ash used.

Grinding Soap-powder.—Mills of various types are used for reducing the coarse lumps or slabs to powder, one form of which is shown in Fig. 139.

Under ordinary conditions an attendant upon the mill is usually required to maintain a continuous feed and to remove the powder as accumulation makes it necessary. The grinding-mill as a rule is subjected to severe usage and to maintain it in efficient operation such matters as lubrication, care of belting, etc., should not be neglected.

Competition in the soap-powder field has led many manufacturers to the use of talc as a cheapening ingredient. This material is easily incorporated with the powder, being added to it in the grinding process. It is without detergent value and fortunately is harmless.

A satisfactory method of handling soap-powder consists in storing the material in a hopper or bin, as it comes from the crusher, and transferring it thence by gravity to the mill as required. This arrangement of crusher, hopper for crushed material, mill and hopper for powder is shown in Fig. 140.

It will be readily seen that the arrangement shown may be varied to suit conditions, so that the crusher may be located on any floor of the building where the framed material is most easily delivered, and the mill may be also located where most convenient.

By using two small elevators, both crusher and mill may be located on the same floor, and the bins placed against the ceiling or on the floor above. In the mill shown in Fig. 139 the material is reduced to powder by repeated blows of rapidly revolving steel beaters running in a special casing. The bottom of the casing is composed of a perforated screen of a special metal, which unites great strength and toughness with phenomenal wear-resisting qualities. The powder is rapidly reduced by the action of the beaters, and the fine material is instantly discharged through the screen. All parts of the casing are easily accessible for examination and cleaning. The only wearing parts are the screens, which are durable and inexpensive. The mill is intended to receive its supply from a storage-bin overhead, and to deliver into one below or into barrels. In Fig. 141 is a diagram for setting up the mill to discharge into barrels. The frame consists of 4-in. dressed yellow pine and is large enough to take a sugar-barrel. The sides of the framework should be cov-

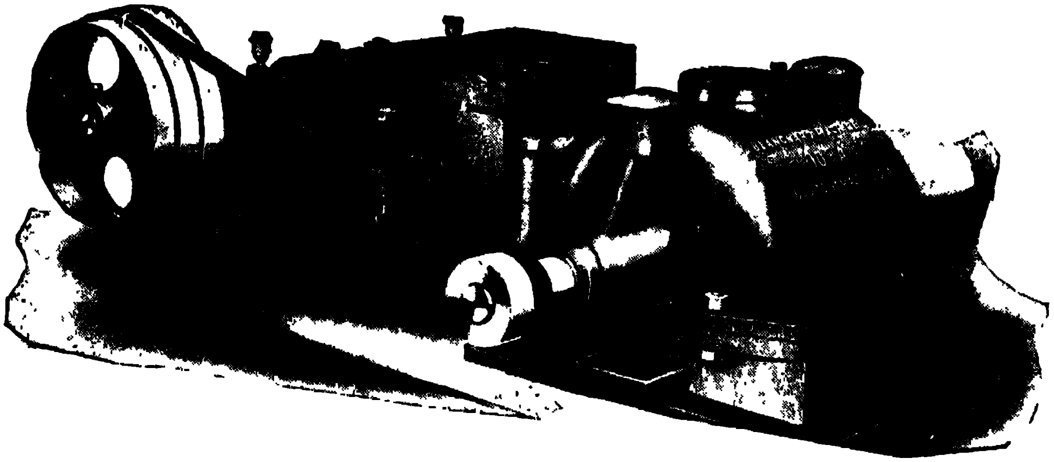


FIG. 139.—Soap-powder Grinding-mill.

ered in with ordinary unbleached cotton sheeting. A door is provided, consisting of a light frame, cloth-covered, which is to be held in place by buttons as shown. The mill draws air through its sides and discharges through the screen. This air circulation is necessary to keep the powder cool.

The cloth sides of the frame allow the air to escape, while retaining the dust.

The mills can be set to discharge into a bin, elevator, conveyor, or any manner desired, but there should always be some such outlet for air provided. In many cases a dust-bag, made of one piece of double-width cotton sheeting sewed together at the edges and made about 6 to 8 ft. long, will serve the purpose. The upper part can be connected by a 3-in. pipe to any convenient part of the discharge-casing, and the lower end should be hemmed and tied together with a heavy cord. When the dust accumulates, untie cord and empty out. Shake dust from sides of bag frequently. If the bag is allowed

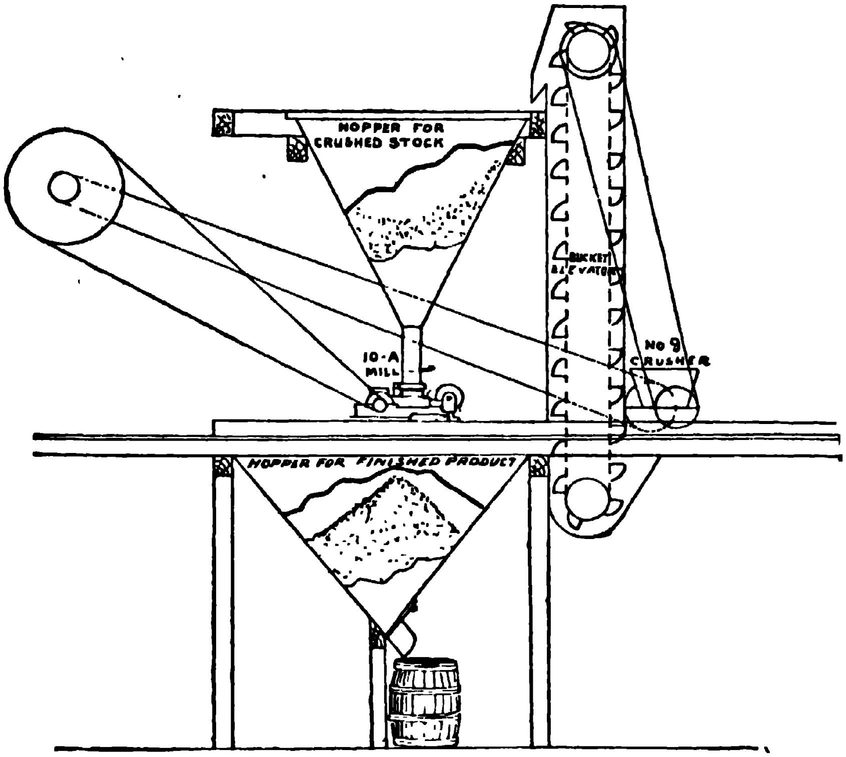


FIG. 140.—Diagram of Soap-powder Plant.

to fill up, the air cannot escape and the machine will be liable to heat up the powder and stop work.

In Fig. 142 is shown a very compact and self-contained device for manufacturing soap-powder which is not without many points of peculiar merit. It consists essentially of a disintegrator or pulverizer which connects by means of an upright duct with a system of sieves contained in the overhead bin from which the powder, reduced to the degree of fineness required, is discharged into the barrel on the floor, or into a bin discharging on to the floor below. The disintegrator consists of a disc bearing cast-steel pegs rotating at the rate of 1500 or more revolutions per minute and contained within a case to both sides of which likewise are attached cast-steel pegs. The disintegration of the powder is thus effected by concussion. The rapid revolution of the interior disc creates sufficient pressure to force the ground material into the sieve-chamber above, where it is deposited on a sieve of given mesh to which a lateral motion is imparted by means of a cam attached to the countershaft. The powder passes through the sieve into a suitable receptacle, while the coarser material with the air is returned by means of a tailing-duct to the disintegrator to be reground.

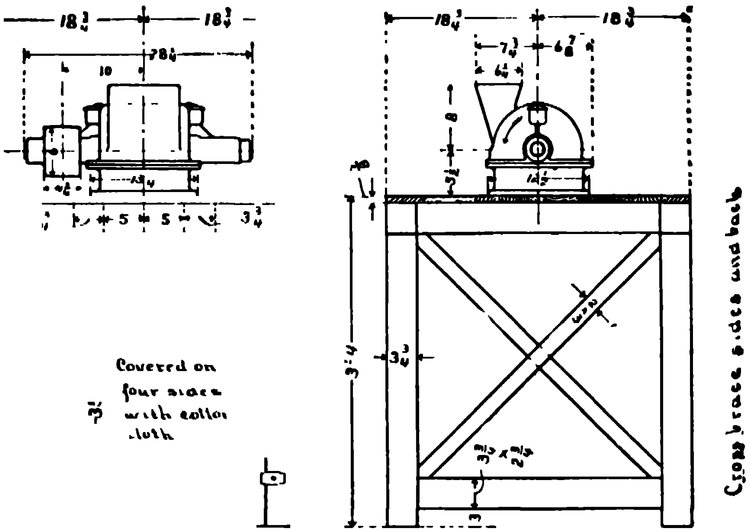

18¾
18¾
28¼
10
5
5
3¾
7¾
6⅞
6¼
8
3'4"
3¾
Covered on four sides with cotton cloth
Cross brace sides and back

FIG. 141.—Diagram for Setting up Mill to Discharge into Barrels.

Packing Powder.—Where large numbers of cartons are to be packed, it is essential to adopt some method to insure that they shall all be filled exactly full and that the cost of filling shall be as low as possible. Many of the larger manufacturers use special weighing and packing machines. The usual method is to determine the

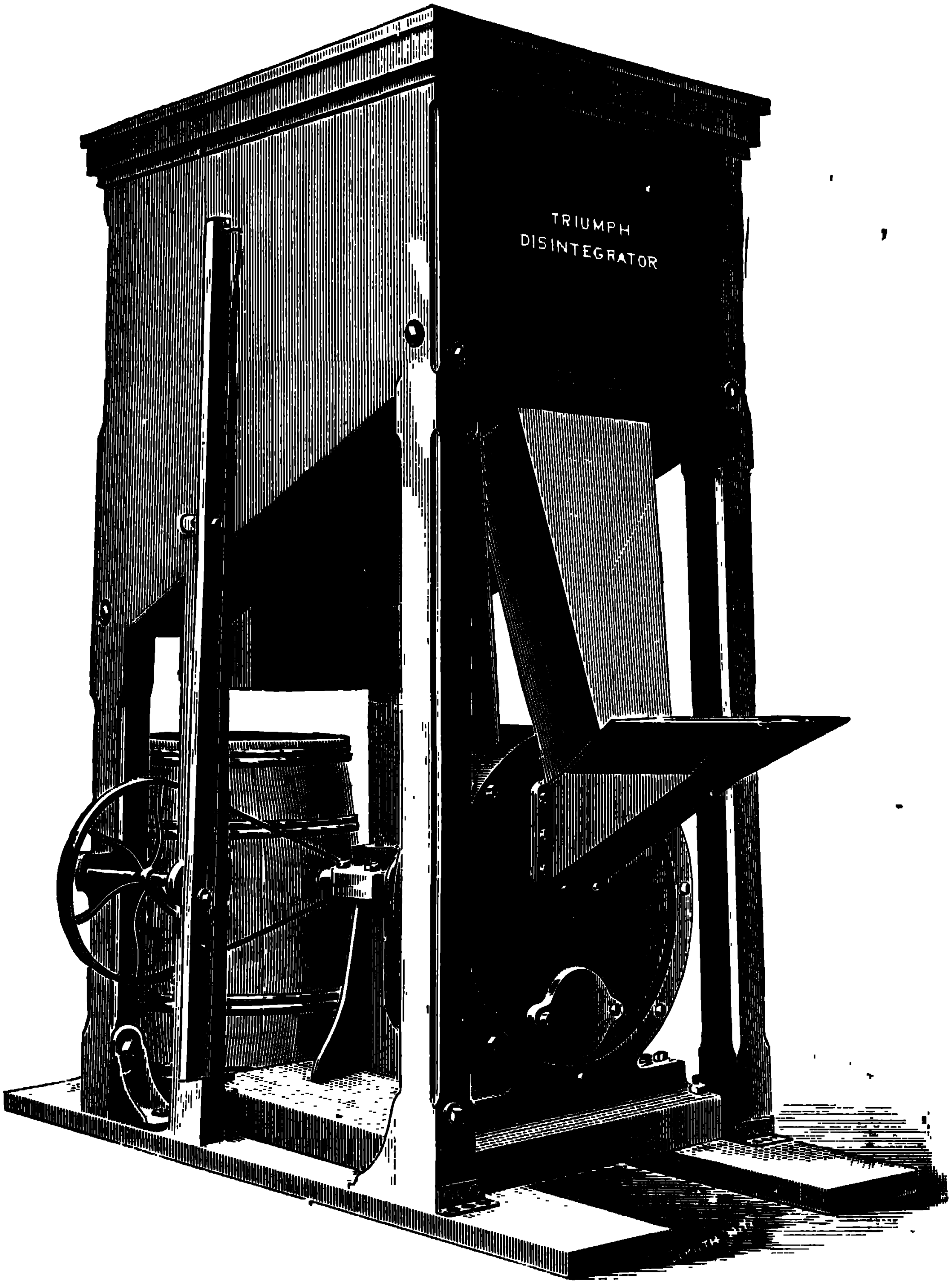


FIG. 142.—Soap-powder Plant.

bulk that the desired weight of material will fill and make the cartons of the proper size to hold this bulk. In this connection it should be borne in mind that there is a difference in the volume that a given weight of powder will occupy, depending on fineness of grinding. Therefore it is best to use powder just as it comes from the mill to determine this volume before ordering the cartons.

A very effective and inexpensive method of filling the cartons is to make a number of tin boxes, open on one end, of proper size to just slip inside the carton and deep enough to contain the required volume of powder. These tins are to be closely packed side by side on a tray as large as can be conveniently handled. The powder can be spread on the open top of the tins and levelled off with a shovel or hand-scoop. One minute's work will fill a whole tray of tins. The tray is then placed on the packing-table, where the packers take the tins one by one, slip a carton over the open top and turn upside down, leaving exactly the right quantity of powder in each carton, and place the empty tin on a new tray ready for refilling.

Continuous Method.—The manufacture of soap-powder on a large scale permits of the fullest opportunity for the introduction of labor- and time-saving devices. The use of an automatic weighing-machine, attached to the soap-powder bin which receives the ground powder from the mill, allows of the discharge of a uniformly regulated amount of powder into the carton or shell. From the crushing of the coarse powder by the disintegrator to the completion of the process the operation as previously described and illustrated is continuous. There is in use where the volume of manufacture permits a device or an arrangement of devices which makes the manufacture of soap-powder an uninterrupted process from the soap-kettle to the pasted package. This arrangement consists essentially of a specially constructed crutcher for mixing the ingredients, a mechanism for cooling, drying, and reducing to a coarse form the material which on a smaller scale of manufacture is framed, cooled, stripped, cut into slabs, dried and disintegrated, a grinding-mill, automatic weighing-machines, belt conveyor for filled packages, and their pasting and packing in cases by specialized labor.

Scouring-soap Base.—As cocoanut-oil is the best stock to use for soap of this character, its high glycerin content (upwards of 13 per cent) makes it desirable to recover it wherever the oil is used in amount sufficient to make the recovery of glycerin practicable. The preparation of scouring-soap base from cocoanut-oil by the semi-boiled process has already been described. Its preparation by the boiled process falls under Class I and is carried out in the same general manner as the preparation of soap-powder base. Owing to the peculiar nature of cocoanut-oil, its manipulation on the stock change is essentially different from that of all other stock with the exception of palm-kernel oil.

Stock Change.—Cocoanut-oil requires for its saponification by the boiled process caustic lye of not less than 20° Bé. and the presence of lye always in considerable excess until after danger of the soap thickening from lack of it or boiling over from sudden and general saponification has been passed. The procedure to be observed on the stock change of cocoanut or palm-kernel oil alone is essentially as follows: Whether stock and lye are added successively or simultaneously is immaterial; the presence of strong lye, either in the kettle or readily available for rapid introduction into the kettle when needed, is the material point. With all the oil in the kettle a considerable proportion of lye at 20° Bé. is added and boiling begun. Saponification will soon ensue, indicated by the mass becoming homogeneous, at which stage 20° Bé. lye is added continuously with vigorous boiling as long as it is absorbed by the oil. If saponification ensues more rapidly than the lye needed is added, the mass tends to thicken, in which event more steam should be admitted and lye added quickly, even to the degree of graining the soap.

Should saponification ensue rapidly with sudden evolution of heat, resulting in the contents rising quickly in the kettle, lye, if it is at the time available, or cold water should be added for the double purpose of cooling the mass and satisfying its affinity for lye. With the danger of thickening and swelling safely passed, the strength of the lye should be gradually increased to 25° Bé., but not in such excess as to grain the soap. Boiling should now be continued as with an ordinary tallow soap until the taste of strength remains permanent. Should it be desired to withdraw a neutral lye, the remaining strength after complete saponification has been assured may be taken up by the addition of a few dipperfuls of cocoanut-oil. Incompletely saponified cocoanut-oil soap, it must be observed, even though no caustic strength be present, has a taste at first difficult to distinguish from that of strength. Cocoanut-oil soap, as has been explained elsewhere, requires considerably more salt to grain than ordinary soap.

Graining.—Where considerable soap of this class is made it may be advisable to grain the first boiling with strong lye up to 35° Bé., and to use the strong stock lye thus obtained on the stock change of a succeeding boil, which is then finished neutral and grained with salt. By this procedure the salt consumption for two boilings is reduced one-half and the content of glycerin in the second neutral stock lye is increased practically 100 per cent.

To satisfactorily grain a cocoanut-oil soap with salt alone sufficient salt must be added to make the waste lye withdrawn a saturated salt solution containing upwards of 25 per cent of salt. And when the lye is withdrawn hot considerable soap is retained in solution which separates as a thick scum on cooling. This is removed and returned to the kettle on the stock or strengthening change. Graining with strong lye is easily effected, it being sufficient to add the lye and boil it well through after each addition until clear lye separates. This lye is removed for subsequent treatment as suggested before. Where salt is recovered its consumption need not be such an item. The two procedures as suggested are equally practicable and their adoption will be determined by prevailing circumstances.

Strengthening and Finishing.—Either of two procedures may be followed on this change: lye at 15° to 20° Bé. in small amounts may be added and boiling continued either with or without the addition of water until the soap closes, showing throughout an unmistakably genuine caustic taste, or water may be added to close the soap, which is then grained with 20° Bé. lye and boiling continued as on the strengthening change. The former procedure is a combination of the strengthening and finishing changes; the latter is the strengthening change. With the latter procedure, after the soap has been strengthened, the lye may be withdrawn and the soap brought to a finish by the addition of water, when it is ready to be used for mixing. Both procedures are equally practicable and the adoption of either will be determined by the thoroughness of saponification on the stock change and the urgency of requirements.

Classes II and III.—The simplest treatment of a grained soap involves merely the saponification of the stock, separation of the waste lye by graining, and the closing of the soap with water or weak lye preparatory to its further utilization. To insure complete saponification a separate strengthening change may be introduced if desired. Soaps made after this general manner are comprised under Class I, the chief members of which are the soap bases for soap-powder and scouring-soap. Such soap is not settled and all the ingredients added to the kettle are removed.

Under Class II are comprised all processes for soap-boiling which require only a stock, strengthening, and settling change. The most simply and quickly made settled soaps are included in this classification. By reference to the outline of the four general classes of processes for making grained soaps, the development of intricacy of manufacture may be readily traced. The difference between the processes included under Class I and Class II is that in the latter the soap is settled and a nigre remains in the kettle, while in the former all the soap is removed. There is less difference between the processes included under Class II and Class III. By reference to the outline on page 289, under Class III, greater thoroughness is secured by the introduction of a wash change with brine, whereby any excessive alkalinity or coloring-matter may be removed.

Influence of Detergents on Textile Fibres.—For a thorough understanding of the character and requirements of textile soaps it is desirable that the soap-manufacturer, who supplies a demand from the textile industry, be familiar with the purpose for which his product is used and the nature of the material to be cleansed. It is also desirable that the professional laundryman be similarly informed, for much of the friction arising between the manufacturer and professional user of detergents may be eliminated by a thorough understanding on the part of the manufacturer of the work to be done with his product, and on the part of the user, by the adaptation of the most suitable material to the fibre or fabric to be cleansed.

There are but four fibres used in the manufacture of articles of personal use and ornament, viz., cotton and linen of vegetable origin, and wool and silk of animal origin. These alone or in admixture form the multiplicity of fabrics in the manufacture and use of which, cleansing, and hence the use of detergents, is essential.

Cotton and linen in a pure state are composed of cellulose, a compound which constitutes the woody fibre of plants. In a raw state they are associated with various amounts of non-cellulose matter, amounting in the cotton fibre to about 5 per cent, which is removed in the process of manufacture. Wool and silk, built up by the vital activity of animal organisms, are of complex composition and in their raw state are associated with secretory substances varying in amount with the fibre: with wool, according to its origin, from 20 to 50 per cent and with silk from 18 to 30 per cent. As the term "detergent" comprises all substances used for cleansing, they may for the moment be most simply classified as alkalis and acids. The chief members of each group will be considered in their influence upon the respective fibres mentioned.

Cotton.—Dilute solutions of the caustic and carbonated alkalis in the cold, or when boiling, or under long exposure do not deleteriously effect the cotton fibre. Concentrated solutions of the caustic alkalis cause the fibre to swell and become transparent and when the alkali is removed by washing the fibre is thicker and shorter. When treated with dilute mineral acids and then well washed, the fibre is unaffected; but if dried without washing, the fibre is made brittle and soon disintegrates. Concentrated acids very materially alter the fibre. Organic acids, being much milder in their acid properties than mineral acids, are less injurious. Chlorine-gas, occurring commercially as bleaching-powder, behaves as an oxidizing agent, with the formation of hydrochloric acid as a secondary product. It should be used only in dilute solutions, for when used in excess, oxidation products are formed with the compound of the fibre, both animal and vegetable, whereby its durability is greatly reduced. As linen is identical in its composition with cotton, its behavior with the reagents mentioned is the same.

Wool.—Both dilute and concentrated solutions of caustic alkalis act injuriously upon wool, tending quickly in concentrated solution, and with long exposure in dilute solution, to dissolve the fibre. Dilute solutions quickly deteriorate the quality of the fibre by causing it to lose its lustre, to shrink and become harsh, and to acquire the disagreeable property of felting, as a result of the forcing apart of the outer scales of the fibre. The influence of alkaline carbonates depends upon the length of the period of exposure and concentration and temperature of the solution, being less harmful with dilute solutions at low temperature and less with potassium than sodium carbonate. Wool withstands well the action of dilute and fairly concentrated acid solutions, although under exposure in concentrated solutions disintegration ensues. Silk, being a fibre of more delicate structure than wool, is considerably less refractory towards the agents mentioned.

Textile Soap.—As soap is a vehicle for free alkali, although the acid salt of the fatty acid is not without beneficial detergent value, it naturally belongs with the alkalis in the classification of commonly used textile detergents. The requirements of a soap for textile purposes is determined by the character of the fibre to be cleansed and the stage of manufacture of the fabric in which the soap is used. As most textile soap is used in the woollen industry, because of the greater foulness of the wool fibre, the discussion of the use of soap in this field will precede a discussion of its use in the cotton and silk industry.

Wool Textile Soap—Scouring Raw Wool.—The encrusting substance of the wool fibre is of twofold character, viz., wool fat and wool perspiration, commonly called yolk, and consisting respectively of mixed ethers of undetermined composition, a portion of which is unsaponifiable, and of potassium salts of volatile and fixed fatty acids with small quantities of sulphates, chlorides, and phosphates of potash. The purpose of wool-scouring is to remove the encrusting matter, which if incompletely removed, or if the wool has been felted or matted, the fibre cannot be satisfactorily dyed, carded, combed, drawn, spun, woven, and finished. The yolk and dirt must be separated with the least injury to the fibre. It is especially desired to avoid high heat and strong alkali. The temperature of the scouring-bath should not exceed 125° Fahr. It is desired to use a soap that will dissolve freely at this temperature. It is generally agreed that any neutral soap free from alkali and uncombined fat, without rosin, sodium silicate, mineral oil, any unsaponifiable substance or other filling material, is adapted for this purpose. A neutral olive-oil potash soap fulfils these requirements, but its high cost militates against its use. It has the least deleterious effect upon the wool fibre and is of milder effect than a soda soap. The natural occurrence of potash salts in wool-fat indicates the superiority of such a soap. A neutral soda soap free from filling, with addition of carbonate of soda, gives good results, the most suitable proportions of soap and soda-ash solution for wools of different quality being determined by experience and careful experiment. Rosin is generally condemned as an ingredient of textile soap; although of considerable lathering power, it is difficult to wash out and traces remaining act as a "resist" to the dye. Soaps made from oils of marked odor, as cocoanut or the semi-drying oils, as cottonseed and corn, are also condemned, because of the persistence of the odor in the finished fabric, which is frequently noticeable in unboxing the goods.

Scouring the Woollen Fabrics.—Subsequently to scouring the raw wool, it is oiled preparatory to spinning, after which the yarn is woven into the fabric. As all oils used on the stock must be removed before finishing, only oils should be used that are easily saponifiable. Any animal or vegetable oil of suitable odor may be used if it can be easily saponified and the resulting soap dissolved at a temperature not exceeding 125° Fahr. Red oil is used almost exclusively at the present time. Its first use for this purpose was considered a great step in advance in textile manufacture, as the cost of oiling was greatly reduced and the subsequent scouring made cheaper owing to its more ready removal. The soap most suitable for scouring the fabric is determined by the quality of the goods and the nature of the material used in oiling. With wool oils of inferior quality a scouring-soap of strong detergent power must be used. Mineral oils are generally condemned for oiling. They are unsaponifiable and are, as a rule, used only for inferior goods. The proportion of soap used for scouring is variable and averages from 15 to 20 pounds per 100 pounds of cloth. When red oil is used for oiling, scouring becomes comparatively easy and simple; with this oil 2 to 2.5 pounds of sal-soda may be substituted for 10 pounds of soap. A good wool oil should supply the fullest lubrication of the fibre with the utmost ease of saponification and removal. The scouring-soap should be made from stock approaching the general character of the oil used in oiling.'

Milling the Woollen Fabric.—The object of the milling or fulling process is to shrink the cloth by raising the outer scales of the fibre, whereby they may interlock and the fabric thus made more compact. No cloth can be satisfactorily handled unless the soap is adapted to its particular needs. With mills turning out a variety of goods, all cannot be satisfactorily treated and finished with the same grade of soap. With high-grade goods of unmixed fibre the most suitable soap is easily determined, but with mixtures of cotton and wool, cotton and worstéd, wool and silk, a soap satisfactory with one fibre or mixture of fibres will be found unsatisfactory with other mixtures. The entire finish and beauty of the cloth depends upon its thorough cleansing. The soap used should be free from alkaline silicates, which lend a harsh feel, and from rosin and semi-drying stock, as cottonseed- and corn-oils, the soap of which is difficult to completely remove and tends to impart cloudiness, odor, and stickiness.

Tallow, palm-oil, or a mixture of these stocks yields a heav-bodied soap of excellent fulling power and fair solubility. Owing to the high percentage of sodium stearate in pure tallow soap, it is less soluble. Palm-oil soap is superior to that made of tallow.

Silk Textile Soap.—In the treatment of the silk fibre, soap is used for two purposes, viz., the separation of the fibre-encrusting matter, or silk glue, from the fibre, the thoroughness of the removal being determined by the quality of goods desired to be made, and in the dyeing process, soaps of the same quality being equally satisfactory for both purposes. A neutral potash soap is commonly used for which olive-oil alone, or in admixture with palm- or cocoanut-oils, is best suited. Owing to the sparing solubility of tallow soap at low temperatures, it is not adapted for silk-scouring.

It is not necessary that the soap should be quite neutral: a slight degree of alkalinity is rather of advantage, for the silk gum is rather more soluble in alkaline solutions than in neutral solutions: but an excess of free alkali in the soap should be avoided, particularly if that free alkali is present in the form of caustic soda, for this may affect the fibroin of the silk, making it hard and dull.

The best soap for silk is a soft soap made from good olive-oil or lard-oil, for such soaps are easily soluble and pleasant in odor. Hard soaps made from the same oils are generally used, particularly the olive-oil soap, for they are cheaper than soft soap. Palm-oil soap, either hard or soft, does well, but is not so soluble as an olive-oil soap.

Tallow soaps are not suitable, and should not be used. Cotton-oil soaps are apt to be colored, while they also tend to become rancid. Palm-nut oil and cocoanut-oil soaps are excellent as regards being soluble and free from color, but they have a tendency to become rancid, which is rather objectionable. Castor-oil forms a very soluble soap, but the odor is rather objectionable. Soaps made from red oil are very good, being easily soluble and free from odor.

Cotton Textile Soaps.—In comparison with the consumption of soap in the woollen and silk industries, its consumptive outlet in the cotton trade is inconsiderable. Cleansing or bleaching of the cotton fibre other than the removal of mechanical impurities is applied only to the fabric. The harmlessness of dilute alkali and acid solutions upon the cotton fibre eliminates the necessity of soap as a mild detergent, so indispensable in the woollen and silk trades. The chief cleansing action to which the fibre is subjected is bleaching, which process is essentially chemical and involves the oxidation or other chemical destruction of the coloring-matter.

Stock for Textile Soap.—Potash soaps made from olive-oil or olive-oil foots and associated in use with carbonate of potash, form a combination that leaves both wool and silk in the finest condition as regards feel and appearance and with least harmful effect upon the color of the dyed or printed fibric whether it be wool, silk, or cotton. The high cost of these materials, with competition, has made substitution necessary, and although the use of the stock mentioned is indispensable for high-grade goods. for goods of inferior quality, especially woollen, cheaper stock is commonly used. Red oil replaces olive-oil, and caustic soda, potash; pearlash is replaced by soda-ash. Cottonseed-oil foots are used largely in the manufacture of the cheaper textile soaps. Olive-oil foots and palm-oil are seldom if ever used unmixed, but merely in quantities sufficient to impart their characteristic odor and color to a straight-tallow or good-grease soap.

The following is an analysis given for a typical mill or textile soap:

Water............	25.36 per cent	From tallow, good grease, palm-oil, olive-oil, or mixture.
Fatty acids........	67.31 " "	
Combined alkali...	7.33 " "	

A specification for scouring-soap could advantageously be as follows: The soap must not contain: (1) more than 30 per cent of water; (2) more than 1 per cent of free carbonate unless otherwise stipulated; (3) it must have no free caustic soda; (4) it must contain no silicate of soda nor rosin soap unless so stipulated; (5) there must be no foreign mineral matter; (6) it must not contain nitrobenzol or other substances to disguise unpleasant odors; the odors should not be disagreeable when rubbed in the palm of the hand.

A specification for a mill soap or a fulling soap could be drawn as follows. The material desired under this specification is a common grade of soap and should meet the following conditions as near as possible: (1) It must not contain more than one-fourth of 1 per cent of foreign mineral matter; (2) it must contain no silicate of soda nor rosin unless so stipulated; (3) it must have no free caustic soda; (4) it must not contain more than one-fourth of 1 per cent of uncombined carbonate of soda unless so stipulated; (5) it must contain no organic matter foreign to a pure soap; (6) it must contain no nitrobenzol or other substance to disguise the odor; the odor should not be in the least disagreeable when rubbed in the palm of the hand; (7) the soap must be made up approximately of 7 per cent combined alkali, 63 per cent fatty anhydrides, and the balance water.

Soap for Printers' and Dyers' Use.—The calico printer wants a soap which is easily soluble in water, hence for this reason palm-, olive-, and cocoanut-oil soaps are to be preferred to a tallow soap, which is not very soluble. The soap must be quite neutral in its properties; if it contains any free alkali, this might act upon the colors and alter their tints or cause them to bleed; alkalinity, however, is avoided by care in making. Rosin, cottonseed-oil, and linseed-oil soaps, from their ready solubility, whereby much alkali is liberated, cause colors to run or bleed, hence the use of those oils should be avoided in making calico-printers' soap. As it is somewhat difficult to make a perfectly neutral soap from cocoanut-oil, the soap-maker takes care to use little or none of that article, more especially as it is not a cheap soap-fat. Printers' soaps should not have any tendency to leave any odor in the goods, or, if so, then this should be a pleasant one. For this reason soaps made from tallow, lard, cotton-seed, and nut-oils are not good ones to use, while palm- and olive-oils make good soaps with rather pleasant odors. It therefore happens that, on account of the reasons here set forth, the calico-printer prefers a soap made from palm- or olive-oils to any other kind of soap, and, although he does not object much to tallow soap, yet he carefully avoids any soap which contains rosin or cottonseed-oil. In preparing cotton goods for dyeing, a wider choice of soaps is available, and any fairly good make of soap may be used; perfect neutrality is not necessary—in fact, a slight amount of alkalinity is of benefit rather than otherwise, as it helps to cleanse the cotton. A soap made from any of the soap-fats enumerated above, alone or mixed together, may be used. Soap is in some methods of dyeing added to the dye-bath, and such soap is required to be of better quality than that used in cleansing the cotton. It ought to be neutral, so that it will not have any action on any colors dyed in the soap liquor; a strongly alkaline soap might alter the shade of chrysamine, for instance; it should not be liable to impart any unpleasant odors to the cloths. For these reasons the best soaps to use are palm-oil or olive-oil, or a soap made from a mixture of those fats. For soaping cotton goods after dyeing, which is, however, not often done for the same reasons as set forth above under printers' soaps, palm-oil and olive oil soaps are those to be recommended. The woollen-dyer only uses soaps after he has dyed his cloths for the purpose of assisting in the milling, and he finds that a good soap made from tallow, or palm- or olive-oil, gives him best results; it should not be strongly alkaline, although a slight degree of alkalinity is of but little consequence, for it is generally neutralized by the trace of acid which is invariably present in woollen goods after dyeing, and this acidity of the goods and the alkalinity of the soap tend to neutralize one another. Before woollen yarns and cloths are dyed, it is usually necessary to remove any oily matter which they may contain. This is done in a soap liquor, and a very common soap for this purpose is one made from bone-fat and cottonseed-oil. Occasionally rosin is added. It is not needful to have a neutral soap; in fact, a little alkalinity is rather desirable than otherwise, as it helps to emulsify the oil in the goods and so makes its removal rather easier. The soap ought to be one which is fairly easily soluble in water, so that it can be readily washed out of the goods after they have been treated, as any left in might tend to produce defects, such as unevenness in dyeing.

Manufacture of Textile Soap.—In consideration of the requirements of textile soap previously discussed, the stock available comprises chiefly olive-oil foots, red oil, grease of good quality, and cottonseed-oil. The resulting soap may be either hard soap made with caustic soda, or soft soap made with caustic potash alone or in admixture with caustic soda. The manufacture of soft soap has been discussed in the section devoted to semi-boiled soap. As neutrality is the prime essential of textile soap, its manufacture falls under Classes II and III. As the combination of stock is variable according to the varying requirements of different consumers, attention at this place will be directed only to the combination of red oil with alkali. In the manufacture of toilet-soap base and of floating soap, the manipulation will be discussed in detail, and as the procedure of operation is the same, the directions applying there apply with equal force to the manufacture of hard textile soap.

Red Oil.—Red oil is the liquid portion of the fatty acids, separated from stearic and palmitic acids, obtained in the decomposition of tallow by either the distillation, autoclave, or Twitchell process in the manufacture of candle-stock. It is of varying composition, according to the process and thoroughness of the decomposition, the non-acid matter consisting of undecomposed glyceride and hydrocarbon oil. Being an acid body it combines immediately with caustic lye of all strengths. This characteristic necessitates a different method of procedure in effecting combination with alkali than is required for neutral fats. Oleic acid, having stronger acid properties than carbonic acid, is able to displace the latter from combination with alkalis, thus permitting its saturation with alkali by means of soda-ash. As red oil is a satisfactory stock for soap-powder base, either alone or in admixture with neutral stock, directions for its combination with caustic soda or soda-ash may be followed in its utilization for that purpose. Red oil, being deglycerized olein, yields no glycerin, hence waste lye from stock changes, in which it is used exclusively, should be withdrawn neutral and discarded. Whether red oil is used alone or in combination with neutral fat of firm body as tallow will depend upon the degree of solubility desired in the finished soap, or in general upon the specifications demanded of any particular textile soap. If less red oil than tallow is used, the latter may be saponified first and the excess of strength remaining may be taken up by the red oil, after the same general procedure as followed in the saponification of tallow and cocoanut-oil for toilet-soap base. We are concerned at this place with its treatment alone either with caustic soda or soda-ash.

Combination with Caustic Soda.—Theoretically oleic acid combines with 14.19 per cent of caustic soda, or with practically it equivalent weight of 20° Bé. caustic lye. The simplest treatment is to add all the lye of this density required for combination to the kettle and then to add, with vigorous boiling as rapidly as it is combined with alkali and uniformly mixed throughout the mass, the red oil to be used. As combination proceeds, should the mass tend to stiffen and retard even boiling, water may be added. If strength remain after thorough boiling, oil should be added in sufficient quantity to take it up. Should this procedure be found impracticable, oil may be added to an excess of caustic lye, and, after combination is under way, oil and lye may be added at the same time, but slowly, and with the latter always in excess sufficient to prevent bunching or general stiffening of the mass. With red oil, soap may be quickly and easily made, and by removing excess of strength by the careful addition of oil, a neutral waste lye after graining may be withdrawn. The ease of combination of red oil with alkali well adapts it for removing traces of strength from waste lye previous to treatment for evaporation.

Combination with Soda-ash.—As oleic acid combines with 14.19 per cent of caustic soda, the following proportion gives its combining weight with sodium carbonate or soda-ash:

$$\underset{106}{Na_2CO_3} : \underset{80}{2NaOH} :: X : 14.19;$$

$X = 18.8$ per cent of soda-ash theoretically required for combination. The combining weights of all fatty acids may be expressed in terms of sodium carbonate by multiplying the percentage of caustic soda absorbed by 1.325. This percentage of sodium carbonate is present in a 24° Bé. solution. The requisite amount of dry ash to saturate a given weight of red oil may be added to the kettle and sufficient water added to dissolve it to form a solution of not less than 30° Bé. Solution is effected by the aid of steam, and when complete the weight of red oil to be saturated by the weight of soda-ash is slowly added, boiling meanwhile. Combination ensues at once with rapid evolution of carbonic acid gas, which combined with too rapid addition of oil or too vigorous boiling, or both, may cause the contents of the kettle to boil over. As combination ensues the mass thickens from the formation of soap and boiling proceeds less evenly. As the evolution of gas ceases, completion of combination is effected by the addition of caustic lye, which will vary in strength and amount by the thickness of the soap. Formation of a neutral soap is effected as already described. Owing to the varying acidity of red oil the estimation of the exact amount of soda-ash required is not practicable and it is not necessary, as whatever neutral oil may be present can be killed only with caustic soda, which is also required to bring the soap to the proper consistency for graining. From the percentage already derived the superior economy from the point of alkali used may be calculated from the following relations: 100 pounds of oleic acid require 14.19 pounds of caustic soda or 18.8 pounds of sodium carbonate for saturation. These are theoretical figures; in practical operations they will be exceeded according to the purity of the materials.

The following practical directions for effecting combination of fatty acids with soda-ash relate particularly to fatty acids prepared by the Twitchell process, but are applicable to fatty acids obtained from any source by any process:

If the deglycerinized fat contain 95 per cent or over of free fatty acids use 19 per cent of 58 per cent soda-ash. If under 95 per cent but over 90 per cent use 18 per cent of soda-ash. Start with an empty kettle and pump into it enough hot 30° Bé. soda-ash solution to supply the above mentioned percentage of ash, or make the solution directly in the kettle by adding the ash to the proper amount of water in the kettle and boil until dissolved. A little less than the calculated quantity of water should be added at the start, so that when the solution has been made and heated to boiling it will not be much under 30° Bé. Bring the soda-ash solution to a strong boil with open steam and run in the fatty acids. Continue adding fatty acids slowly and boil vigorously, so as to get rid of the gas given off as quickly as possible. If the soap rise high in the kettle, try stopping the addition of fatty acids for a few minutes, but continue boiling vigorously. If the soap drop, the addition of fatty acids should be continued. A point, may, however, be reached, generally when about three-fourths of the fatty acids have been added, when it will be necessary to boil more gently to keep the kettle from boiling over. It is, however, desirable to boil as vigorously as possible without boiling over from the start until all the fatty acid is in and all the soda-ash taken up and the gas expelled.

After the ash has all been taken up and the gas expelled, caustic is added and the soap boiled until it no longer takes up caustic, just as would be done in killing stock with caustic in the regular way. The soap may be settled immediately after the saponification, or, what is better, given a salt change and strengthening change and settled in the usual manner. Such soap will be identical in every respect in washing properties, filling properties, and appearance and keeping qualities with soap made from the same stock by caustic.

It is very important in saponifying with ash that no more ash than the stock will take up be used, and that the boiling be thorough enough before the caustic is added, not only to use up all the ash present, but also to drive out all the carbonic-acid gas liberated. If caustic be added while soda-ash is still unconsumed in the kettle, the soda-ash will be lost. Also if there be any gas unexpelled when the caustic is added it will take up an equivalent amount of caustic to form carbonate, which will be a loss.

Rosin may be previously dissolved in the stock and saponified with it or it may be saponified separately, using ash, and pumped in the kettle after the saponification of the stock has been completed.

By the above method there is no difficulty in saponifying a charge for the largest sized kettles used in a half day if the stock is supplied as it should be, while small kettles take a shorter time and in all cases the time is but little longer than would be required to stock the same kettle in the usual way.

Of course fatty acids can be saponified with caustic the same as the neutral stock if desired, the only difference being that the lye must be put in the kettle first and the stock added, the same as would be done in saponifying red oil, which is a fatty acid.

Fatty acids are especially well adapted for making soft soaps with potash, thus recovering all the glycerin, by the preliminary process, which is retained by the soap when neutral fats are used.

Castile Soaps.—Genuine castile soap is made by settling a pure olive-oil-soda soap, but little of which is made to-day owing to the high cost of olive-oil. Olive-oil is replaced either partially or completely by peanut-oil, cottonseed-oil, and light-colored red oil. Tallow also may be used in varying amount up to the degree that the characteristic slimy lather of soap made from olein-stock may not be destroyed. Olive-oil foots is the basis of the so-called green castile soap, which is likewise replaced in varying amounts by the same substitutes for olive-oil. The pale-green color of the soap may be heightened by the use of suitable organic coloring-matter. Soap of this kind is used for toilet purposes and is not, as a rule, filled. So-called castile soap is practically identical in composition and methods of manufacture to the olive-oil soda soaps for textile purposes. Stock proportions for a good imitation castile soap comprise 65 per cent of olive-oil and 35 per cent of tallow. For green castile soap olive-oil foots may be replaced by tallow in amounts varying from .25 to 75 per cent; in the latter case chlorophyll compound may be used to restore the color of a straight foots soap. In their behavior toward saponifying agents olive-oil and olive-oil foots do not differ greatly from tallow. The procedure of manufacture is described in the section devoted to toilet-soap base.

Marine or Salt-water Soap.—The specifications for salt-water soap issued by the Bureau of Supplies and Accounts of the Navy Department of the United States require that such soap must be well made from pure cocoanut-oil and the necessary alkalis only. It must be entirely soluble in both sea-water and fresh water, and make a good lather in using, and must be free from filling of any kind. When received, the bars shall weight 3 avoirdupois pounds each and shall contain:

Carbonated alkali (equivalent to Na_2CO_3) between 2 per cent and 3 per cent;

Free alkali (equivalent to $NaOH$) not more than 0.50 per cent;

Salt ($NaCl$) not more than 3 per cent;

Mineral matter, including silicate of soda, sulphate of soda, etc., not more than 0.50 per cent;

Water not more than 55 per cent;

Balance to consist exclusively of cocoanut-oil combined with the proper amount of alkali to form a neutral soap.

This soap may be made by the semi-boiled process, as described in the preparation of cocoanut-oil soap for scouring soap, without recovering the glycerin. By this process it may be transferred directly to the frame and crutched by hand until sufficiently cool. Filling not above the proportions limited by the specification may be added to the kettle and incorporated by boiling. The high glycerin content of cocoanut-oil makes it desirable, wherever this stock is used in sufficient quantity, that the glycerin be recovered. This may be done after the same general procedure outlined before for abstracting the glycerin from scouring-soap base. Soap made for mixing with silex is finished much thinner than is possible for satisfactory settling preparatory to framing.

The stock change for marine soap is practically as has been described for cocoanut-oil soap-base for scouring-soap. The soap may be grained with alkali or salt and the stock lye withdrawn with strength or neutral. If with strength, the same may be recovered, according to the quantity present, by either working the lye over on the stock change of a subsequent boiling or by boiling it up with sufficient stock to combine with it, whence the lye is run to the storage-tanks. If grained with alkali, sufficient caustic may be mechanically retained to render necessary the addition of but little strong lye on the strengthening change. After removing the strong stock lye, the soap is boiled up, either with or without the addition of much water, according as the water of condensation may suffice to close the soap. As the soap closes and becomes neutral 18° to 20° Bé. lye is added until the soap is open, and boiling is continued throughout the day. After prolonged boiling, should the soap tend to close with excess of strength present, saturated pickle, or brine, should be added. Steam is then turned off and the lye allowed to settle out. The pickle serves to keep the soap open when no caustic lye is required and facilitates the separation of the strength. The strength lye is withdrawn to storage-tanks or to another kettle to be worked over with fresh stock.

Cocoanut-oil soap cools quickly and becomes very firm. The degree of hydration to be effected on the settling change to satisfy the requirements of the specification, viz., not more than 55 per cent of water, must be determined by experience. After the proper consistency has been obtained by the addition of the requisite amount of water and boiling, the soap may be framed at once and crutched in the frame until stiff. If filled (the restriction of carbonated alkali to 2 to 3 per cent corresponds to the addition of 8 to 10 per cent of 36° Bé. soda-ash solution) the soap is run directly to the crutcher and cooled, and filled, according to the specification, and is then framed. This soap is ready to strip on the day after framing. Cocoanut-oil soap permits of a large amount of filling either of detergent matter as carbonate and silicate of soda or of inert matter as alkaline chlorides.

Boiled-down Soap.—Soft stock, as red, corn-, and cottonseed-oils, is not adapted when used alone to the manufacture of unfilled, grained, and settled soap, to be described, owing to the softness of the finished product and its resulting wastefulness in use. This softness may be counteracted by the addition of suitable filling, as saturated soda-ash solution and sodium silicate, to the soap after having been finished very coarsely in the settling change, and also by depriving the soap, previous to framing, of a portion of the associated water. The latter procedure yields the boiled-down soap. This modification of the grained process is, therefore, adapted to the utilization of very soft stock as red oil, soft grease, cottonseed-oil, cottonseed-oil soap-stock, and rosin in combination. Considerable soap of this quality was formerly made, but now only in diminishing quantities and for certain classes of trade. While a good durable soap from suitable admixture of firm and soft stock, or from soft stock of good quality alone, may be made according to this procedure, in present practice, soft and generally inferior stock is commonly used. Such soap has been almost completely displaced by filled settled rosined soap. All boiled-down soap is characterized by short texture. It is generally cut and stamped without pressing. With the exception of milled soap, but little dehydrated soap is now made, whether for household or toilet purposes, the tendency being toward a product which combines most satisfactorily a maximum degree of hydration and firmness of body. Attention will be directed first to so-called "oleine" soap, which is a firm-bodied soap made from soft stock and rosin.

Procedure.—The general method of Class I is followed until the completion of the strengthening change. The use of rosin necessitates an extra rosin change, the procedure of which is described on page 338. The combination of red oil with caustic soda or sodium carbonate is described on page 318. The procedure of strengthening is described on page 340.

After withdrawing the strengthening lye, water is carefully added with thorough boiling until the soap is brought to the consistency of a soft, flat grain. Should an excess of alkali remain it may be removed by either the addition of sufficient cocoanut-oil to absorb it or by the introduction of a pickle change, whereby it is removed in the pickle lye.

Water of condensation from the live-steam coil may suffice, without the further addition of water, to bring the soap to the desired finish. Where soap of this quality is made the kettle should be provided with a closed steam-coil. With soft stock making a thin soap and cooling slowly, a mottle does not form. After the desired consistency on the finish has been obtained, the soap, after a few hours' settling, is ready to be framed. It may be run directly to the frame and hand-crutched or to the crutcher, as with settled soap, where it is cooled and then framed. If made in large amount and filled with either sodium carbonate or sodium silicate or both, it is necessary to cool it to 140-150° Fahr. and subject it to the same treatment as a settled rosin soap. Much sodium-carbonate solution is not advisable, owing to the readiness with which soap made from soft stock sweats and effloresces.

The dehydration, according to the procedure described, is not effected by boiling down as much as it is by not adding the water required to settle the soap in the ordinary way. Genuine boiled-down soap is very firm soap from soft stock subjected to boiling on brine, whereby the water is abstracted from the soap and transferred to the brine. The density of the brine determines the degree of dehydration of the soap.

After settling out the pickle, the soap is framed pure and crutched by hand until cool, although the power crutcher may be used as well.

By the use of the crutcher for cooling the soap is made homogeneous at a low temperature, whereby the characteristic appearance arising from slower cooling by hand is destroyed.

The Mottle.—The term "mottled," as applied to soap, refers to a characteristic discoloration of the natural body of the soap arising from the incorporation of mineral salts of characteristic and varied color and produced by their exclusion from the firmer soap into softer soap during the progress of cooling. The same effect may be produced to a degree in cold-process soap by adding a small amount of mineral coloring-matter of the color desired to the soap before the period of spontaneous heating and working it through by dexterous use of the hand crutch. The mottle in this case is more of a streak than is the characteristic mottle produced by selective separation as in the ordinary mottled soap.

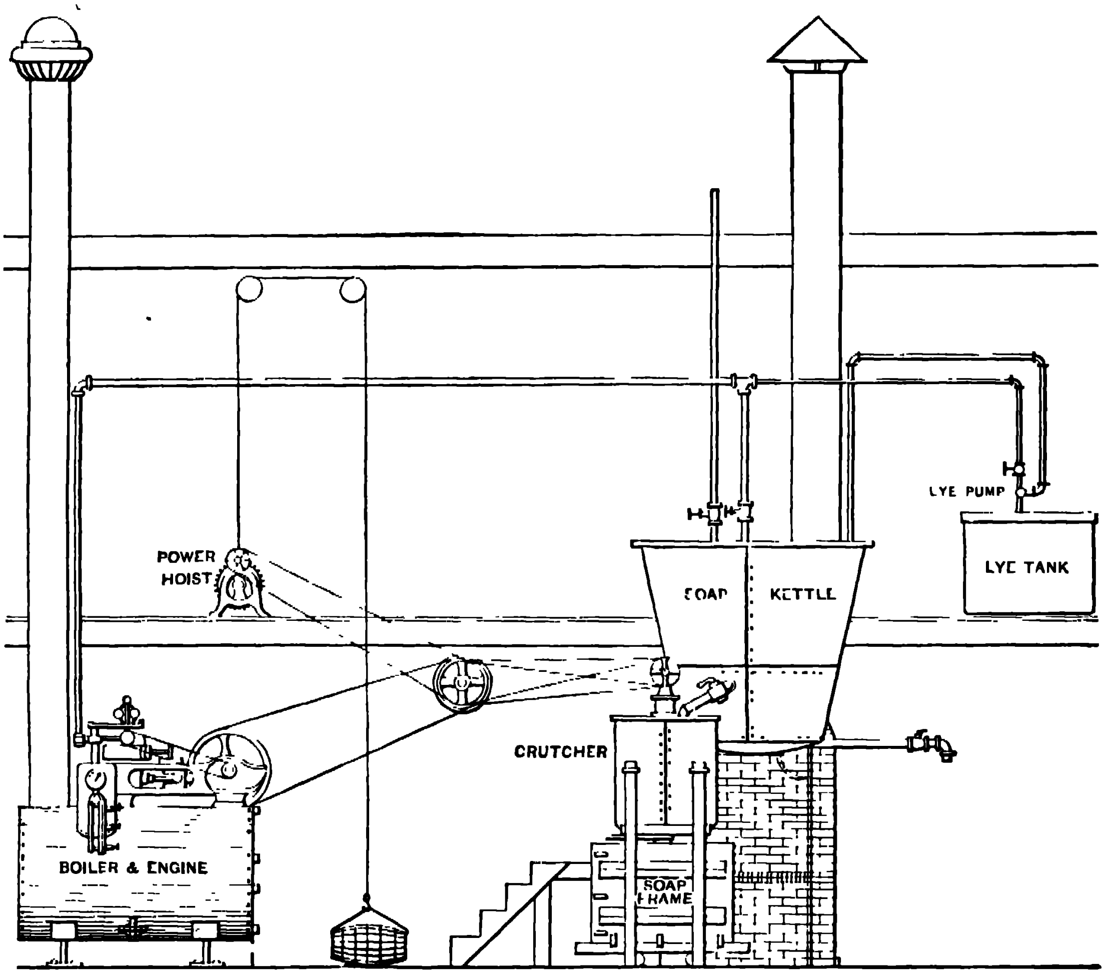
Mottle is the result of crystallization, which is a process of purification whereby all foreign matter, as far as conditions allow, is expelled from the crystallizing or solidifying body. In the formation of mottle this expulsion takes place from soap solidifying at a higher temperature into soap solidifying at a lower temperature, whence further expulsion is impossible and the foreign coloring-matter remains on cooling mixed with the softer soap. This behavior will be more pronounced the more fluid the soap and the slower the cooling. Under these conditions a coarse mottle ensues. With the soap more open, it is less fluid, cools more rapidly, and as a result the selective separation occurs only slightly, if at all, forming a general discoloration or only a fine mottle. With the intermediate condition, wherein the soap cools neither too slowly nor too quickly and is of stock of the best proportions of stearin and olein, whereby the desired conditions of fluidity and temperature are best attained, the characteristic appearance known on cooling as "mottle" most satisfactorily develops.

Mottled Soap.—The procedure employed in the manufacture of genuine mottled soap is the same as for ordinary settled soap up to the settling change. Beyond this the modification employed for mottled soap is of very limited application in American practice. There is no better process for the manufacture of general purpose soap than the grained, settled process, and any departure from it is in the direction of sophistication and inferiority of product. For toilet purposes a mild neutral soap is desired, likewise for textile purposes, while for domestic use, a soap whose natural detergency is increased by the addition of 8 10 per cent at the maximum of combined carbonate and silicate of soda solutions satisfies the requirements. Where greater detergency is desired than may be effected by the soaps mentioned, soap powder and the carbonated and caustic alkalis are available. Where for toilet and domestic use, detergents inferior to those mentioned find sale, the intelligence and purchasing capacity of the consumer are low, and which market the reputable manufacturer with pride in his products seeks only as a last resort.

The mottled or marbled appearance of soap is a vestigial characteristic of early days of the industry marked by primitive methods and the use of impure material. Soap as a manufactured commodity did not then exist as it does to-day. With social progress and the growth of improved industrial methods, materials, and competition the mottled appearance which the consumer had associated with purity, notably in the case of castile soap made from olive-oil and impure caustic soda, was retained but produced by different means in cheaper material. In American practice the native stock was limited in variety, being at first exclusively tallow and later, as the demand for special soaps increased, cocoanut-oil was added. Previously the value of rosin as a stock ingredient was demonstrated, and in very recent years cottonseed-oil has become extensively used. The tendency has been to limit the variety of stock available ,and its general quality has been lowered by the more remunerative outlet of the better grades, in other industries.

In European practice vegetable stock, notably olive-oil, was early used, and in later years, owing to the greater cost and less abundance of animal fats, the variety of stock of vegetable origin has increased. More vegetable stock is used abroad in proportion to animal stock than in domestic practice. As the mottle was first produced with oil stock, its persistence has been confined to soap made from that stock and to soaps purporting to be made from it. And as the amount of vegetable oils used in the United States is very small compared with that of tallow, together with the use of rosin and the high hydration of the finished product, mottled soaps for household purposes have never been popular in the United States. Manufacture of the old-style mottled soap requires working with comparatively small batches. With kettles of immense capacity as are now common in the United States, the satisfactory incorporation of inert salts to produce a mottle would overtax the ingenuity of the most skilful soap-boiler. In Germany, where this type of grained soap is extensively made, conditions of production are quite different from those prevailing in the United States, there being upwards of 3000 separate soap-boiling establishments in the German empire. These are necessarily small and the production of small batches of large yield is the rule.

Unfilled Mottled Soap.—Soft stock is killed, rosined, if rosin is used, and strengthened as with an ordinary settled soap. If it be desired to purify the soap by the partial separation of a nigre, water is carefully added and boiled through until proper conditions for the separation of impurities by settling are obtained. Unfilled soap, likewise heavily rosined soap or soap of any kind made from soft stock, should invariably be settled with a coarser finish than is given soap intended to be filled or any soap made from firm stock. After standing for the requisite time—a day or two may suffice—the nigre is pumped out from below into another kettle. To the remaining soap is added 25° Bé. brine and the whole well boiled through. The purpose of the pickle change is to withdraw the excess of water added to the soap during the previous settling change for the separation of nigre. The amount of pickle needed and its density will depend upon the amount of water to be removed from the soap, and the consistency or grain in which it is desired to leave the soap for upon this depends the proper development of the mottle on the cooling of the soap in the frame. If the soap is left too open, it will not have the right degree of fluidity and will cool too quickly to allow of the proper dissemination of the mottle throughout the mass. If left too close, the soap will be too fluid, will cool too slowly, and the coloring-matter will be uniformly distributed throughout the mass without the development of a distinct mottle.



Courtesy of Houchin and Huber.

FIG. 143.—Central American Plant for Mottled Soap. Boiling Either with Steam or Direct Fire.

After the removal of the pickle lye the proper condition of the grain may be obtained by the addition of water, when an excess has been withdrawn the lye. It is thus seen that the proper conditions for mottling may be obtained by boiling a closed soap on pickle to withdraw water, or by adding water to a soap already grained.

Mottling Agents.—With impure materials, mineral salts and natural coloring-matter were sufficient to produce the mottled effect. It may be emphasized by the use of insoluble mineral colors Soluble colors should not be used, as they disseminate too readily. The most commonly used dyes are venetian red, ultramarine blue, yellow ochre, and copperas, or ferrous sulphate. The last gives a greenish mottle which on exposure turns red from the oxidation of ferrous to ferric sulphate. The proportions to be used vary from 4 to 8 ounces per 100 pounds of soap. The quantity of dye for the soap to be mottled is thoroughly mixed with a portion of soap from the kettle, after which it is returned to the kettle and the whole boiled through. The soap is then run into frames and crutched by hand to 130° to 140° Fahr., or until after danger of the mottling agent settling to the bottom of the frame has passed.

Soap framed in the open condition peculiar to mottled soap is very compact and has a short, brittle texture, and, unlike a settled soap, will not press satisfactorily. It is, as a rule, cut into bars of various shapes and weights and stamped.

Filled Mottled Soap.—Cocoanut-oil soap possesses the property of assimilating before separation large amounts of solutions of common salt, sodium carbonate, and sodium silicate. This property is likewise possessed by palm-kernel oil. This stock mixed with tallow in the proportion of one part oil and two parts tallow and saponified with strong lye at 25° Bé. in small batches by the semi boiled process can be made to incorporate as high as 30 per cent of silicate of soda and water, making a yield of about 200 pounds of product from 100 pounds of stock. By reversing the proportions of oil and tallow or by eliminating tallow entirely the capacity of cocoanut-oil soap to assimilate alkaline salts will permit a total yield of product of over 300 pounds. The addition of a mottling agent to the mass of soap and salts in the kettle before framing permits of the formation of a characteristic mottle on cooling in the frame.

CHAPTER IX.

SETTLED ROSINED SOAP.

Class IV. Rosined Soap. Requirements of Household Soap. Stock. Preparation and Use of Caustic Lye. Stock Change. Stock Lye. Rosin Change. Rosin Lye. Strengthening Change. Strength Lye. Settling Change. Nigre. Utilization of Nigre. Killing Rosin with Soda-ash. Duration of a Boil of Soap. Crutching. Filling. Operating Crutcher. Framing. Yield of Soap.

Class IV. Rosined Soap.*—The term "rosined" as applied to soap has reference to the fact that at a certain stage in the manufacture of a soap from tallow, or mixed tallow and oil, commonly cotton-seed-oil when soft stock is used, rosin in a solid state is added to the kettle and combined with caustic soda, the resulting compound called sodium resinate being incorporated with the soap and imparting to it its characteristic properties. Rosined soaps constitute the greater proportion of all soap made and sold in English-speaking countries. While a few States in the southeastern part of the United States enjoy a practical monopoly of the production of rosin, the English were the first manufacturers of rosined soap. Americans, however, greatly improved it, and, after the close of the Civil War, during which the production of naval stores was prostrated, demonstrated the value of rosin as an ingredient of soap for domestic or household purposes. At this time the use of a saturated solution of sodium carbonate, to impart a firmness to soft-bodied soap and to increase its detergency, was introduced—first, it is claimed, by A. Van Haagen in Philadelphia. The improvement in the appearance and quality of rosined soap by the incorporation of varying proportions of soda-ash solution greatly increased its popularity. Owing to its superior detergency and cheapness it soon replaced tallow soaps of inferior quality made at that time by very primitive methods. At this time sodium silicate came into general use by the larger manufacturers and came soon to be regarded as an essential ingredient of rosined soap.

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* See outline of the four general classes of processes for the manufacture of grained soap, page 289.

Rosin in no sense can be regarded as an adulterant of soap. On the contrary, notwithstanding what objections may be associated with its use, it is a valuable and, for soaps for many purposes, an indispensable ingredient. Its compound with alkali, while not a true soap, possesses marked detergent properties. Its cheapness, its soft body when combined with alkali and the ready solubility of this compound in cold and hard water, and its ease of combination with alkali and incorporation with true soap, whereby the firmness and comparative insolubility of the latter are reduced, make rosin an essential ingredient of all soap for domestic use. It is unsuited for use, however, for all soaps where light color is desired, likewise for textile soaps, as explained in the section devoted to that subject.

Requirements of Household Soap.—For general domestic use a soap is required that dissolves readily, i.e., lathers freely in both hard and soft water, that contains no free alkali, and that does not contain alkaline filling in excessive amount. By an excessive amount of alkali may be understood both free and combined alkali present in the soap in such proportions that with the use of the soap the hands, fabric or color of the fabric, or any object cleansed in the general course of household work may be more or less injuriously affected. White settled soap made from tallow does not fulfil these requirements although made of superior stock.

The purpose for which a soap is intended should determine its composition; the purpose having been determined, such admixture of stock should be selected as will permit the highest detergency and economy in use with the least injury to the user or the object cleansed. Such a soap may be made of tallow in admixture with grease or cottonseed-oil (price permitting) and rosined from 90 to 50 per cent, according to the quality desired; the whole thoroughly saponified, settled, and incorporated with 6 to 8 per cent of 36° Bé. soda-ash solution and silicate of soda. By reference to the outline of the general processes of manufacture of grained soap the final process is that applicable to the manufacture of rosined soap. While soap made by this process is the cheapest in point of cost, it represents the other extreme in skill required to produce from given materials an article of the highest quality.

Stock.—The quality of the stock used is determined by the grade of soap desired to be made; the proportions of different stock equally available from the point of quality are determined by the relative prices of competing stock, it being endeavored with soap of a definite standard of quality to maintain that quality at the lowest cost. As rosin is the characteristic ingredient of this grade of soap, the glyceride stock is selected on the basis of the percentage of rosin it will carry with the maximum brightness and firmness of the finished product. As the general effect of rosin is to soften and darken the soap, the color and firmness of the glyceride stock should be in inverse proportion to the quantity of rosin used. As a general rule, however, in practice the quality of the glyceride stock is shaded to the percentage of rosin carried and the selling price of the finished product. Tallow and grease with varying proportions of cottonseed-oil, according to the price of the latter, form the glyceride stock commonly used. In some brands of rosined soap cocoanut- and palm-kernel oil are used. The grade of rosined soap produced is determined by the quantity and quality of rosin used. From 90 to 40 per cent of rosin on the basis of glyceride stock represent the common extremes. WG or WW rosin should be used when the percentage is large. As a rule it is better to rosin good stock high, quality of the finished product permitting, than to rosin inferior stock low. With lower grades of rosin the color rapidly increases, the soap being thereby greatly deteriorated in this respect.

Preparation and Use of Caustic Lye.—Caustic-soda lye is best prepared from commercial caustic of 74° grade, the caustic requirements for a given kettle charge being conveniently estimated for all practical purposes by the proportion of a given weight of stock (stearin, olein, and palmitin) requiring an equivalent weight of 20° Bé. lye. It is by no means necessary that all the lye needed be prepared at this density. It is, as a rule, diluted in the pipe by water from a convenient water connection as it flows from the caustic melting-tank to the soap-kettle, the proper adjustment of lye and water for any given density being readily ascertained by experience.

In the preparation of caustic lyes of different densities from various grades of caustic, the influence of the impurities, chiefly

TABLE XXI.—SHOWING THE PERCENTAGE OF SODA, Na_2O, IN A CAUSTIC LYE AT THE TEMPERATURE OF 60° FAHR. AND THE QUANTITY OF MIXED FATS WHICH MAY BE SAPONIFIED BY THIS LYE. (TUNNERMANN.)

Specific Gravity.	Degrees Baumé.	Per Cent of Soda Na_2O.	Quantity of Mixed Fats which May be Saponified by 100 Parts of Lye.	Specific Gravity.	Degrees Baume.	Per Cent of Soda, Na_2O.	Quantity of Mixed Fats which May be Saponified by 100 Parts of Lye.
1.05	6	3.626	33	1.2392	27	15.11	139
1.0587	7	4 231	39	1.2453	28	17.714	145
1.0675	9	4.835	44	1.2515	28.5	16.319	151
1.0764	10	5.44	50	1.2578	29	16.923	156
1.0855	11	6.044	55	1.2642	30	17.518	162
1.0948	12	6.648	61	1.2708	30.5	18.132	167
1.1042	13.5	7.253	66	1.2775	31	18.73	173
1.1137	15	7.857	72	1.2843	31.6	19.341	179
1 1233	16	8.462	78	1.2912	32.4	19.945	184
1.133	17	9.066	83	1.2982	33	20.55	190
1.1428	18	9.67	89	1.3053	33.5	21.154	195
1.1528	19	10.275	95	1.3125	34	21.758	201
1.163	20	10.879	100	1 3143	34.2	21.894	202
1.1734	21	11.484	105	1.3198	34.5	22.363	206
1.1841	22	12.088	111	1.3273	35	22.967	212
1.1948	23	12 692	117	1.3349	36	23.572	217
1.2058	24.5	13 297	122	1.3426	36.7	24.176	224
1.2178	25	13 901	128	1.3505	38	24.78	229
1.228	26	14.506	134				

Calculated on a chemically pure basis.

sodium chloride, sodium carbonate, and sodium sulphate, is to reduce the active value of the solution for the specific gravity indicated. This reduction in saponifying power is least for the highest grades and greatest for the lowest as a natural result of the increased percentage of these impurities present. There has been arranged in the table on page 97, Table XI, the percentage of sodium hydrate present in lyes of different densities, made of the usual grades of caustics, corresponding to the densities of lye made from chemically pure caustic. It is assumed in the common specific gravity tables that the total alkali is present entirely as caustic, which never actually occurs. The figures stated for reasons previously given are generally slightly higher than would be found in practice. In the Appendix appears a table calculated on the basis of the actual caustic alkali commonly occurring in the commercial grades of caustic which for accuracy is more to be relied upon than one calculated on a chemically pure basis.

The density of lye used depends upon the character of the stock, the nature of the change, and the stage of the change. With the stock commonly used for rosined soap, lye above 15° Bé. at the beginning of the stock change should not be used; saponification will not only proceed with difficulty but it will be necessary later to add water to create conditions necessary to the easy boiling of the mass. The various grades of tallow work best with an initial density of not over 15° Bé.; cocoanut-oil, 20° Bé.; cottonseed-oil and similar neutral stock require a density of 12°-15° Bé. An excess of caustic lye retards saponification by graining the mass, in which condition combination cannot ensue. Lyes of suitable density saponify easily because the soap dissolves as fast as formed, the mass passing quickly from an emulsion to a clear paste.

Stock Change.—With mixed tallow and cottonseed-oil stock the tallow should be added first and killed and the excess strength taken up with the oil. Tallow or grease is received at the factory either in barrels or by tank-car. In the former case it may be run by gravity directly from the stock steamer to the soap-kettle and lye at 15° Bé. added simultaneously. When received by tank-car it is first weighed and pumped to a storage kettle, which may be either the soap-kettle in which it is to be utilized or a separate tank or kettle, from which it is run either by gravity or pump into the saponifying-kettle. Saponification may be started by either the simultaneous or successive addition of stock and lye. With stock and lye at 15° Bé. running in, steam is turned on and the mixture boiled vigorously. After saponification has once begun the lye should not be added in such excess as to retard it, but its addition should be adjusted to the volume of tallow added, so that with constant boiling it will be taken up by the tallow almost as fast as added. In the saponification of two fats that differ in their ease of combination with caustic lye of a given density, one saponifying best with a weak lye and the other with a strong lye, they may be added either simultaneously and killed with a lye of intermediate density, or the most easily saponified stock may be added first and killed, increasing the density of the lye as saponification proceeds and towards the close leaving considerable excess of strength in the kettle to be exhausted by the more difficultly saponifiable stock. Should for any reason the supply of lye be deficient, as for instance an interruption in its addition, or a quickening of the rate of saponification, the soap will stiffen often throughout its entire mass and require prolonged boiling to melt and reduce to a satisfactory state This occurs most frequently with rapidly saponifying stock, as cocoanut and palm-kernel oils and to a less degree with tallow stock. This sudden, general saponification may be retarded by the quick addition of strong caustic lye or pickle and vigorously boiling it through. With ordinary care in the use of neutral animal stock and the more difficultly saponifiable vegetable oils as cotton-seed-oil, little danger need be apprehended from the contents of the kettle boiling over; but with cocoanut-oil, which saponifies very readily with strong lye with evolution of considerable heat and resulting swelling of the contents of the kettle, care must be exercised lest the very fluid soap boil over. This tendency may be checked by shutting off steam and adding water or strong lye, the purpose being merely to cool the soap or locally grain it, thereby permitting the egress of steam. After saponification is well under way and the greater part of the stock and lye have been added, the density of the lye may be gradually increased. With all the stock in and absorption continuing, lye is added at 20° Bé. in small amounts, with gentle but thorough boiling after each addition. If after successive additions of 20° Bé. lye the strength remains fairly permanent, but in no great excess, boiling should be continued slowly until it is absorbed.

In the phraseology of the kettle-room, "killing stock" is liberating glycerin from combination in a fat and introducing the soda of the caustic solution in its place. The saponification of tallow is a process requiring three stages for the complete elimination of the glycerin and combination of the soda with the fatty acids. While these stages do not admit of exact definition in the soap-kettle, they manifest themselves in certain characteristic conditions which are familiar to every soap-boiler, viz., the emulsion formed on admixture of soap and lye, the pasty mass obtained on steady boiling, and, lastly, the final condition resulting from boiling the pasty mass with an amount of lye sufficient for complete saponification. By means of chemical formulæ, we are able to graphically represent the three successive stages of saponification.

RAW MATERIALS.

Stearin, $CH_2O(C_{18}H_{35}O)$ $CHO(C_{18}H_{35}O)$ $CH_2O(C_{18}H_{35}O)$	Caustic soda, $3NaOH$

I. EMULSION.

CH_2OH $CHO(C_{18}H_{35}O)$ $CH_2O(C_{18}H_{35}O)$	Soap, $C_{18}H_{35}ONaO$ Caustic soda, $2NaOH$

II. PASTY MASS.

CH_2OH $CHOH$ $CH_2O(C_{18}H_{35}O)$	Soap, $C_{18}H_{35}O \cdot NaO$ Caustic soda, $NaOH$

III. CLEAR SOAP.

Glycerin, CH_2OH $CHOH$ CH_2OH	Soap, $3C_{18}H_{35}O \cdot NaO$

On the completion of saponification the contents of the kettle have assumed a clear homogeneous mass in which is present everything that has been added during this stage, viz., soap, glycerin, water, some caustic soda in excess, and some salt, sodium carbonate, and sulphate introduced as impurities with the caustic lye, together with whatever mucilaginous matter or animal tissue that may have been present in the stock. If saponification has been complete, there will be no free or unsaponified fat.

Physical conditions indicating completion of saponification are easily recognized and comprise the entire absence of a greasy feel to the soap when cooled and pressed between the fingers; it should be of short texture and not stringy, and on cooling dry, and easily pressed into chips. In the utilization of stock lye for glycerin, which we shall see later contains the greater part of the glycerin removed during a boiling of soap, it is better to leave the soap with a small amount of free stock and have the stock lye neutral than to have the stock completely saponified and such excess of caustic in the stock lye that it is necessary to work it over with fresh stock to recover it.

With uniform treatment of successive boilings the soap can be brought to a certain condition with a fairly constant but small amount of unkilled stock and with no free alkali in the stock lye. For the maximum separation of glycerin on the stock change it is recommended that the density of lye used throughout the change should not exceed 15° Bé. and the soap grained with dry salt. The volume of waste lye formed with caustic lye of this density is claimed to contain the maximum amount of glycerin. With the stock completely killed and an excess of alkali present, the same may be neutralized by the addition of a sufficient amount of fresh stock or rosin to neutralize it. While it is desired to kill the stock as thoroughly as possible on the stock change, if it involves the presence of strength in the waste lye in excess of 0.4 per cent, the treatment suggested should be followed. The stock change is primarily to effect combination of the stock with caustic soda whereby the elements of water are absorbed and glycerin is set free, which on subsequent graining is discharged into the stock lye, which should be free from caustic alkali. Therefore the absorption of the last portion of alkali required should be effected with care. Organic reactions proceed more slowly than do inorganic reactions, especially towards the close, and require intimate admixture, effected in this case by prolonged boiling, to make them complete.

Graining.—At the close of the stock change the soap should be without turbidity and free from stringiness, due to the presence of unkilled stock, and should fall from the inclined paddle in clear transparent flakes of short texture. The body of the hot fluid soap depends upon the nature of the stock used and the amount of water present. With a given degree of hydration, tallow forms the firmest soap and is most easily grained; cocoanut-oil the most fluid soap and the hardest to grain; olein stock as cottonseed-oil is intermediate. A tallow and cottonseed-oil soap will be satisfactorily grained with 7–10 per cent of salt in the stock lye; a 50 per cent cocoanut-oil soap with 15–20 per cent; graining a 100 per cent cocoanut-oil soap with salt alone is very unsatisfactory, as the grained soap is highly contaminated with salt and considerable soap remains dissolved in the hot lye, which separates on cooling.

In experiments made to ascertain the behavior on saponification of a mixture of stearic, oleic, and palmitic acids towards an amount of alkali insufficient for their complete saponification it was found that there was no selective absorption. This fact, however, is not applicable when soap made from these acids is grained, for their respective solubilities, not only in water but in salt solutions, differ. With the stock thoroughly killed and the soap neutral, it is ready to grain. Dry salt is shovelled in and uniformly scattered over the surface of the soap, boiling the soap well up to the top of the kettle meanwhile. The amount of salt required is soon ascertained by experience as well as by the appearance of the soap. After each addition of salt it should be well boiled through and the appearance of the soap noted. As water is withdrawn from the soap to dissolve the dry salt, it loses its transparency and assumes a grain-like consistency, the individual "grains" being as large as a bean or as small as wheat-seed, according to the amount of salt added and water withdrawn from the soap. Twenty thousand pounds of killed tallow require about 8 or 9 bags (200 lbs. each) of fresh salt. On the first appearance of lye separating the addition of salt should cease and the contents of the kettle well boiled through to secure intimate admixture of the waste lye. When the separated lye runs clear and with no separation of soap on cooling, the soap is sufficiently grained. It is desired to effect the separation of the maximum amount of clear lye with the least amount of salt; this requires thorough boiling of the soap so that the salt and pickle may be intimately mixed with the soap and the maximum dehydrating effect obtained. Steam is now turned off and the lye allowed to settle out. The length of time required for the separation of the waste lye is variable; two hours may suffice, but it is customary to devote the entire day to the stock change and allow the lye to settle out overnight. On the following morning the stock lye is withdrawn and either pumped to the glycerin refinery or reserved in storage until it is convenient to utilize it.

The percentage absorption of alkali of various grades by the different stocks most commonly employed in American practice is shown in the following table.

The variation in the amount of alkali absorbed by different fats and oils, likewise the variation in the amount of alkali absorbed by any particular fat or oil, arises from differences in the composition

TABLE XXII.—POUNDS CAUSTIC SODA OF DIFFERENT GRADES REQUIRED TO SAPONIFY 100 POUNDS OF STOCK.

Soap-stock.	77½ Per Cent.	76 Per Cent.	74 Per Cent.
Tallow	13.7-14.1	13.97-14.37	14.35-14.77
Cotton-oil	13.6-14.0	13.87-14.28	14.24-14.67
Cocoanut-oil	17.5-17.7	17.84-18.05	18.32-18.54
Olive-oil	13.6-14.0	13.87-14.28	14.24-14.67
Palm-kernel oil	15.7-17.7	16.01-18.05	16.44-18.04
Palm-oil	14.0-14.4	14.28-14.69	14.67-15.09
Rosin	12.1-14.0	12.34-14.28	12.68-14.67

Soap-stock.	72 Per Cent.	70 Per Cent.	60 Per Cent.
Tallow	14.74-15.18	15.17-15.62	17.70-18.22
Cotton-oil	14.64-15.07	15.10-15.50	17.57-18.09
Cocoanut-oil	18.84-19.10	19.37-19.60	22.61-22.88
Olive-oil	14.64-15.07	15.10-15.50	17.57-18.09
Palm-kernel oil	16.90-19.10	17.39-19.60	16.12-22.88
Palm-oil	15.07-15.50	15.50-15.95	18.09-18.61
Rosin	13.03-15.07	13.40-15.50	16.00-18.09

of the glycerides themselves, and from the varying proportions in which the glycerides occur in any particular sample. We know that fats and oils are indefinite mixtures of various glycerides, and the amount of alkali absorbed is influenced by the nature of the glycerides characteristic of the stock. The amount of alkali required for neutralization decreases as the molecular weight of the glyceride increases, hence we find that those commercial fats and oils in which glycerides of low molecular weight occur possess the highest alkali absorption. The increased quantity of salt required for graining such stock is due to the presence of those glycerides of low molecular weight whose greater solubility in brine of the sodium salt is a marked characteristic. As the glycerides increase in molecular weight the solubility in brine of the soap obtained therefrom diminishes, hence less salt is required for graining. In Table XLVIII, page 679, is shown the saponifying power of caustic lyes of different densities, made from chemically pure and from the ordinary grades of caustic occurring in the market, in terms of tallow. By reference to this table the amount of stock that can be saponified by any quantity of lye of the density and quality stated may be readily determined within limits of accuracy suitable for all technical purposes to which the table may be applicable.

Stock Lye.—From analyses of stock lyes withdrawn from a tallow-cottonseed-oil soap the following average analysis may be accepted as representing high efficiency of practice.

	Per Cent.
Total alkali, calculated as NaOH	0.37
Salt	9.10
Glycerin	6.50

The total alkali in a stock lye of this character is almost entirely carbonate, present as an impurity in the commercial caustic, indicating that all the active strength has been withdrawn from the lye. With insufficient graining the stock lye on cooling in storage-tanks will separate soap varying in amount with the deficiency of salt. The volume of stock lye withdrawn is indicated by the depth of subsidence of the soap in the kettle.

Rosin Change.—The handling of rosin always involves more or less inconvenience, its dust and stickiness being a fertile source of annoyance. The barrels may be brought in the kettle-room and the rosin broken up in front of the kettle, or it may be reduced in the yard and conveyed to the kettle in cars or trucks, or to a floor over the kettle into which the entire contents of the car are discharged at once, or where the amount used is great it may be reduced to uniform fragments over a grating, through which it falls either into a car or into the boot of a belt conveyor, which conveys it to a horizontal conveyor running over the kettles, into any one, of which it may be discharged at will. (See Fig. 48) After withdrawing the stock lye, steam is admitted to the grained soap and rosin is added by any of the methods above suggested. If added by hand the addition of rosin continues, as a rule, for several hours, during which time only sufficient steam is needed to keep the soap hot and slowly to melt the rosin. With all the rosin in the kettle a considerable amount of lye at 20° Bé. is now added and boiling vigorously begun. Combination ensues immediately and continues rapidly. Lye at 20° Bé. is added as required, a deficiency being indicated by lack of strength in lye, the soap tending to close and by uncombined rosin distributed through the mass. Toward the end of the change, with rosin almost completely killed and sufficient caustic lye in excess, should the soap tend to close from long boiling with live steam, whereby water is introduced, dry salt is added in amount sufficient to keep the soap open. Throughout the entire change the mass should be kept open, first with 20° Bé. lye until no more is absorbed and finally with dry salt. With the soap open the combination of rosin is facilitated by greater intimacy of contact with the lye and a more thorough discharge of the coloring matter is practicable. Rosin, being an acid body, will combine with caustic lye of any density, but for greatest ease and quickness of operation, a strength greater or less than 20° should not be used at any time on the change under normal conditions.

The degree to which the soap is grained may vary, but this does not effect the combination of alkali with rosin as it does with glyceride stock; the former is a direct saturation, the latter is an interchange of the reacting bodies with the absorption of water and with the formation of two dissimilar substances.

To effect combination quickly and not unduly to prolong the change, the fragments of rosin should not be larger than a man's head, otherwise prolonged boiling for melting is necessary. Care should be taken that all the rosin is melted and combined. With deeply dished kettles rosin may sometimes lodge in the cone and resist the action of heat. The rosin change is most easily carried out and produces the darkest lye. It is made with but a small part of the swelling that always attends the saponification of glyceride stock. With the soap open and lye with marked but not excessive strength and with the assurance of the absence of uncombined rosin, steam is turned off and the rosin lye allowed to settle out. The time required for the rosin change depends upon the weight of rosin killed and the quickness with which it is added to the kettle. Under ordinary conditions the rosin is added during the forenoon and the change satisfactorily completed in the afternoon.

Rosin Lye.—According to the procedure described for the rosin change the rosin lye is withdrawn with strength, which is a more or less general practice at present and formerly was entirely so. The rosin lye is highly charged with impurities, which when withdrawn with strength have to be encountered when the lye is worked over to recover the strength. Moreover, its removal for purification involves care and expense. The amount of glycerin as based on the percentage content and volume of the lye is not large. A modification of the procedure of boiling at this stage may be effected with equally satisfactory results from the standpoint of both the soap boiler and glycerin-refiner as follows:

Stock Change.—Kill the stock throughout the change with 15° Bé. caustic lye. Grain with dry salt and withdraw the lye neutral.

Pickle Change.—Add sufficient water to just close the soap, boiling meanwhile, and grain with dry salt. The purpose of this change is to wash out the glycerin mechanically retained by the soap after withdrawing the stock lye. Both stock and pickle lye are bright in color and neutral.

Rosin Change.—Kill the rosin as already described, but leave the soap open on salt alone, with entire absence of strength. Discharge this highly colored and neutral lye to the sewer. By introducing the pickle change between the stock and rosin changes, the glycerin otherwise discharged into the impure rosin lye is removed in the clean pickle lye. This arrangement may be described as a compromise between the soap-boiler and the glycerin refiner, inasmuch as the former desires to effect the maximum discharge of impurity into the waste lye, while the latter desires to receive the lye neutral and with a minimum amount of impurity. Again, the pickle lye may be dispensed with and the rosin lye withdrawn neutral as above suggested, but saved for glycerin. The essential ingredients of such a lye are indicated by the following average analysis:

Alkali, NaOH	0.26	per cent
Salt	9.00	" "
Glycerin	4.50	" "

As shown, no more salt is required for graining than on the stock change.

Strengthening Change.—The strengthening change is an auxiliary change in that it serves to complete the work of previous changes. It is impossible to withdraw neutral waste lye and leave completely saponified stock. Even where no pains are taken with the causticity of waste lye, much stock escapes saponification as shown by the alkali absorption of the strengthening change. The strengthening change also serves as a wash change, for much color is discharged by the alkaline lye. Treatment on this change, as with all others, varies with different soap-makers and also with the same one according to conditions. To the grained soap, after withdrawing the rosin lye, sufficient water may be added, boiling meanwhile, to just close the soap; 20° Bé. lye is then added with constant boiling until the soap is again opened and boiling is continued, with soap maintained in this condition by the addition of 20° Bé. lye as fast as it may be absorbed and the soap tends to close. Lye of greater strength should not be used unless the presence of water in the kettle or a considerable addition of the same by condensation from the live-steam coil may serve to dilute the lye, but even with these conditions a density of 20° Bé. suffices. An excessive amount of lye at 20° Bé. should not be used. It should be added gradually as rapidly as absorbed, and when the absorption ceases the strength present should not be greater than what is required for keeping the soap open. Towards the close of the change with sufficient strength present for the requirements of absorption, should the soap tend to close upon prolonged boiling, it should be kept open by the addition of pickle the density of which is determined by conditions of hydration in the kettle. With much water and the soap thin, 20°–25° Bé. brine may be used; with the soap thick and boiling explosively, brine at 10°–12° Bé. may be used. Scrap from previous boilings should be added on this change, preferably at first to insure melting and thorough incorporation.

To the grained soap at the start, caustic lye at 12° 15° Bé. may be added without the addition of water and the open condition may be maintained as already described. The combination of the last traces of reacting bodies, particularly where one or both are of organic origin, proceeds slowly and often best in concentrated solutions. In the case in question the condition requiring concentration is supplied by the use of strong lye.

The density of the lye added, the degree of graining, whether lye of low density is added directly to the grained soap and the whole boiled until closed, the use of live steam or closed steam, or both, etc., are matters that vary with each operator. No dry salt should be added on this change; one purpose of the change is to leach out the salt from preceding changes retained mechanically by the soap. The soap should be boiled until the excess of strength is permanent and with the soap well open, when steam may be turned off and the strength lye allowed to settle out. Under ordinary conditions one day at least is required for the change.

Strength Lye.—An average strength lye from settled rosined soap made according to the procedure described will analyze as follows:

Alkali, NaOH	6.38	per cent
Salt	4.32	" "
Glycerin	3.30	" "

Absorption of caustic on the strengthening change means the saponification of residual free stock and liberation of a corresponding amount of glycerin, but practically all the glycerin was liberated on the stock change, yet it has been present in considerable amount in each waste lye subsequent to the stock lye, indicating how tenaciously it is retained by the soap. The strength lye is of the least volume of the three waste lyes, in this respect standing third. The stock lye is of largest volume and contains not only the highest percentage content of glycerin, but also the most in absolute amount. The rosin and strength lyes in volume and absolute amount of glycerin present stand in the order mentioned. It is withdrawn usually on the morning following the strengthening change and is either held in storage or added at once to another kettle for treatment with fresh stock to recover the strength, or added to the grained nigre or stock change of other boilings, where it assists in the saponification of fresh stock.

It is not so dark as the rosin lye, but yet very highly colored.

Settling Change.—The character of treatment on the settling change determines the success of the boiling. Deficiencies on previous changes may be corrected on a succeeding change, or by repeating the change, with but little trouble, but with the settling change any deficiency allowed to pass until it is recognized only when the soap is crutched means not only the loss of time involved in cooling but the trouble and expense involved in heating to the boiling-point a kettle of cooled soap and of removing the excess of water by graining.

After withdrawing the strength lye, the open soap is boiled up with the addition of water until it is closed. Free alkali may be present in such amount that with prolonged boiling the soap refuses to close, in which event steam should be turned off, the soap allowed to settle for an hour or so, and the strong lye withdrawn, after which boiling is continued with the addition of water until the soap closes. Additions of water should be made now very carefully and only after each addition is well boiled through and its effect noted. The degree of hydration on the settling change is determined by the quality of stock used and the quality of soap desired to be made. With soft-bodied stock, as grease or cotton-seed-oil, or tallow stock heavily rosined, or firm stock, as tallow and cocoanut-oil, the soap of which is to be unfilled, as castile soap, milled soap-base, and floating soap, the settling should be coarse, with but a comparatively slight degree of hydration. Soap to be filled with soda-ash solution, of recognized hardening effect, as tallow stock lightly rosined, may be settled finer or thinner, i.e., more heavily hydrated than the various kinds of soaps just mentioned. With the conditions of settling reversed, the nigre will be large and the yield of good soap small.

With any excess of free alkali sufficient to prevent closing removed as described and water added with continued boiling until the soap is joined or closed, additions of water are made as rapidly as it is incorporated, the soap meanwhile swelling up in the kettle and becoming stiffer after each addition of water is taken up. With stock thoroughly saponified, there will be no absorption of alkali on the settling change. The soap should be tasted from time to time to observe the causticity. If after the addition of much water the strength has been absorbed and there is a slight tendency to stringiness, strong lye, according to the degree of hydration of the soap, should be added. Should this condition appear early weak lye should be added, but not in such excess that that which remains, after satisfying the demand for lye, cannot be neutralized practicably with cocoanut-oil.

Addition of water should cease when the soap boils up regularly with but little steam and rolls over with a stiff surface, breaking into placques, or saucer-like depressions, which retain their firmness and transparency from the centre to the circumference of the kettle. The appearance which properly settled soap displays on boiling is characteristic and well defined, but requires experience at the kettle, with the manipulation of steam and the addition of water, to produce. Should the addition of water be excessive, the firmness of the soap necessary to produce the appearance described is destroyed and the surface is flat, in which event it is necessary to grain the soap with strong lye, 35°-40° Bé., sufficient to just separate lye. After settling out this lye, the operation of settling is repeated. However, a flat surface does not always indicate an excess as much as an incomplete assimilation of the water. With continued boiling the soap may regain its firmness.

If it be desired to finish the soap neutral, cocoanut-oil may be added toward the close and well boiled through. Not only is the free alkali effectually neutralized, but the appearance of the soap in the kettle, as well as in the finished form, is thereby greatly improved. Well-settled soap should glide from an inclined trowel in broad, tenacious, transparent flakes, without shortness—indicating poor joining—or thinness and stringiness—indicating an excess of water.

It is desired on this change to leave the soap in such a degree of hydration—indicated by its physical appearance in the kettle and when taken upon a trowel—that on standing for a period sufficient to cool the soap to a temperature of 140°-150° Fahr. it will separate into two distinct portions, viz., good soap and nigre. During this period, the length of which is determined by the volume of soap, its degree of hydration, and the temperature, or the season, the contents of the kettle arrange themselves in the order of their specific gravities, the soap tending to rise to the top, whereby the percentage of soap in that portion of the contents of the kettle is increased and the impurities, being heavier, tending to subside. As a result of this interchange, the more highly hydrated soap with the impurities is in the lower part of the kettle and the firmer soap is in the upper part, both portions with a good settle being separated by a sharp line of demarcation.

Nigre.—Satisfactory separation of impurities, chiefly organic matter derived from the stock and rosin and not completely separated by graining, and the relative amounts of good soap and nigre depend primarily upon the hydration of the soap. In introducing conditions essential to the formation of nigre, as noted before, there are two extremes, viz., an excess of water to such a degree that the nigre comprises almost all the soap, and a deficiency of water, whereby the soap not being completely joined the contents of the kettle on standing and cooling resolve themselves into two portions, viz., a little lye at the bottom, and soap the lower portion of which is more discolored, but the whole mass open. It is aimed by the addition of water to strike the medium of these two extremes, but approaching the latter more than the former. And the latter extreme should be approached to a greater degree with soap made from soft stock, or intended to be unfilled, than it is with soap made from firmer stock. The maximum separation of impurities with the minimum volume of nigre, which is simply heavily hydrated soap highly charged with impurities, can be determined only by experience. The volume of nigre naturally varies, as a rule, between 25 and 35 per cent of the total contents of the kettle. The length of the settling period required for the formation of nigre and the rate of its formation are difficult to determine, but are entirely included within the time required for the soap to cool to a temperature suitable for either framing directly or the incorporation of filling by crutching and then framing.

Utilization of Nigre.—The nigre contains all the impurities not discharged into the waste lye withdrawn after the stock, rosin, and strengthening changes and not retained by the soap. It contains practically all the alkali retained by the soap after the withdrawal of the strength lye. On transferring to the crutcher the last frame of good soap, the nigre is ready to grain. The line of demarcation between good soap and nigre is sharply defined, but, as a rule, it is necessary to dip the last two or three frames of good soap, otherwise, by pumping or allowing the good soap to flow out by gravity, currents are created whereby, as a result of the greater fluidity of the nigre, it is mixed with the good soap to the detriment of the latter. Steam is turned on and the nigre boiled up. As it rises in the kettle dry salt is thrown in and well boiled through. Soap in the nigre should be reduced to a very small grain in order to separate most completely the coloring-matter. As a result of the greater amount of water in the nigre more salt is required than for an equal volume of soap. When well-grained the steam is turned off and the mass allowed to subside. When it has reached its original level the lye may be run off into the sewer, as it is worthless. The utilization of nigre from settled white soap, as milled-soap base and floating soap, is described in the following chapter. Where several grades of rosined soap are made, nigre from the best grade is incorporated into a boiling of soap of the next lower grade and the nigre from this grade into a boiling of soap of still lower grade, where, after it has been worked over two or three times, it is transferred to a kettle and allowed to remain until other nigres from the same grade of soap may be added until a sufficient amount accumulates to make a separate boiling. The nigre from this grade of soap may be used indefinitely. The utilization of rosined-soap nigres where several grades of rosined soap are made involves no difficulty, but where only one or two grades of soap are made it must be removed or utilized in some way at frequent intervals, otherwise the color of the soap deteriorates. If it is from a high-grade rosined soap containing not over 50 per cent of rosin on the basis of the glyceride stock, the nigres may be worked into soap-powder. A lower grade of rosined-soap nigre, as well as this grade, may be used for tar soap, the color and odor of the tar effectually disguising the origin of the soap-base. Where only one grade of rosined soap is made, nigres from different boilings may be mixed and settled, the good soap being returned to a boiling with fresh stock. If not too highly rosined the nigre from nigres thus treated may be utilized in soap-powder either alone or in admixture with firm stock. Although rosin alone is not a desirable ingredient of soap-powder when it is mixed with firm stock as described, its deliquescence is greatly reduced. When found that it deteriorates the color or quality of the powder the proportion used may be reduced.

When the nigre is incorporated into a succeeding boiling of soap the stock change may be made upon the nigre in the kettle in which it was grained. Under such conditions the procedure of saponification does not differ from that already described on the stock change. The frequency with which a nigre may be worked over depends upon the color desired in the finished product. Two or three times is the usual number.

With the introduction of suitable conditions on the settling change, and within the limitations of temperature, the longer a well-settled soap is allowed to stand the smaller will be the nigre and the purer the soap above it, with the hydration gradually increasing as the nigre is approached where it reaches its maximum. Analyses of well-settled soap and nigre indicate the soap and water content in the latter to be almost the converse of what it is in the former.

The following analysis of a normal nigre indicates its general composition:

Water..................................	64.32	per cent
Soap (anhydrous).......................	29.04	" "
Alkali (free), NaOH....................	0.52	" "
Alkali (combined), Na_2CO_3..............	2.17	" "
Undetermined...........................	3.95	" "

If on the settling change the soap is left with a slight taste of strength, during the settling period the alkali with what mineral impurities, chiefly salt, may remain, subsides, so that the percentage of free alkali in the soap increases as the nigre is approached. The length of the settling period varies with the volume of the soap, being shortest with the smallest volume. It is likewise affected by the seasons being longer in the summer than the winter and longer with kettles protected against loss of heat by radiation. Too rapid cooling should be avoided, as there then may be introduced conditions analogous to crystallization. The settling period is shorter with firm than with soft-bodied stock, owing to more rapid cooling at all seasons. With kettles yielding 40–50 frames under ordinary conditions, the settling period for all stock at all seasons ranges from 7 to 11 days from the day of settling to the day of framing.

Although the softness of the stock, hence the greater solubility of soap made from it, may affect the volume of the nigre, the influence of this factor is inconsiderable in comparison with the manipulation and the conditions of settling already noted. It has been demonstrated by the chemical examination of soap and the nigre formed from it that there is no appreciable selective separation of soft from firm soap.

Killing Rosin with Soda-ash.—Combination of red oil with soda-ash has already been described. As rosin is likewise an acid body the practicability of saturating it with soda-ash instead of with caustic soda is suggested. Without doubt considerable saving is possible by using soda-ash instead of caustic soda, but practical difficulties intervene to mar the calculation. Rosin soap is of very soft body, it is grained with more or less difficulty, and while these disadvantages are by no means insuperable obstacles, the practice of killing it with soda-ash has never become common. It would seem desirable to eliminate once and for all the color possible to be discharged into a waste lye from rosin soap before the soap is mixed with tallow soap. To work rosin separately so that it may be grained with any satisfaction its body must be hardened. This may be done by killing it with soda-ash on a nigre and with the addition of scrap. The product may then be incorporated with a tallow soap after the stock change in a separate kettle and the whole strengthened as on a regular boiling. In the evolution of methods of soap-boiling the tendency has been towards the utmost simplicity and ease of operation consistent with the best results in the appearance of the finished product. The separate killing of rosin with soda-ash and its subsequent incorporation with a tallow soap seems to be a method to try but not to adopt. The tendency of glyceride stock to swell during saponification demonstrates the impracticability of killing the regular tallow charge upon a nigre and rosin soap base.

Duration of a Boil of Soap.—The time required for making a boiling of settled rosined soap depends primarily upon its size, i.e., the amount of stock and rosin killed. It is believed that the highest economy of operation is secured when each change can be started and completed in a single day. With kettles of average size, allowing one day for each change and from seven to twelve days for settling, an average of two weeks are required before the soap can be taken out. Allowing three days for solidifying in the frame and from two to three days, according to facilities of drying and the state of weather, for cutting, drying, and pressing, upwards of three weeks are required from the first treatment of the raw material to the shipment of the finished product. In the manufacture of milled soap the period of operation intervening between the soap-kettle and the soap-press can be reduced to a few minutes, but the product is considerably drier and more durable, which qualities are not desired under present conditions by the manufacturers of low-priced detergents. Clearness, smoothness, and utmost transparency of structure with brightness of color, and the highest possible hydration consistent with firmness, are the ends sought. Under these conditions any reduction of time involving a sacrifice of these ends will be slow of acceptance.

Crutching.—After the period of cooling, which for the contents of any given kettle is determined by experience, whether the soap has cooled sufficiently to frame may be determined by withdrawing a sample in a bucket from the swing-joint-pipe outlet and testing it with a thermometer. During the settling period the soap has cooled from that temperature at which it was when in contact with steam during boiling to 150° Fahr., the winter temperature, or to 140° Fahr., the summer temperature. The most suitable temperature for crutching depends upon the firmness of the stock and the nature and amount of filling material to be added. With firm stock lightly filled, the temperature of crutching may be higher than with soft stock lightly filled or firm stock heavily filled. In general, settled rosined soap should not be removed from the kettle at a temperature higher than 150° Fahr., for in most cases the soap as it flows out increases in temperature. In summer the crutching temperature should be 10°–15° Fahr. lower than in winter. Soap crutched too hot will separate filling in the frame no matter how smooth it may appear in the crutcher, and from poor incorporation of the filling the soap will effloresce and soda-crack on aging, with resulting great deterioration in appearance and marketable quality. With unfilled soap the temperature may be higher than with filled soap in accordance with the firmness of the soap. With soap of this character it is aimed to withdraw the good soap from the kettle at the temperature that will permit its entire removal after the shortest necessary period of settling and to reduce the temperature to that point at which there will be no separation of the more highly hydrated soap from that containing less water, on subsequent standing in the frame. Soap containing a considerable proportion of cocoanut-oil, as was seen, cools more quickly in the kettle and also more quickly in the frame than does soap made from stearin and olein stock. With well-settled unfilled soap of the former character crutching may be unnecessary, it sufficing to run the soap directly to frames by a system of overhead gutters provided with outlets over each frame, as shown in Fig. 80.

The conveyance of soap to the crutchers from the kettle may be by pump or by gravity. In the latter case, which is simpler and involves less trouble, the top level of the crutchers should be below that of the nigre. With the bottoms of the kettles and crutchers on the same floor, proper conditions for the gravity discharge of hot soap from the kettle are supplied. With crutchers located at a higher level than that of the nigre or removed from the kettles by a distance that makes troughing impracticable a pump is necessary. Unless with a large number of crutchers the pumping is intermittent and often makes much trouble by requiring frequent steaming out of the chilled soap from the soap-line.

The far better plan under all circumstances is to run the soap by gravity from the kettles to the crutchers. With this arrangement hot soap may flow continuously into a box or tank from which the crutchers are filled alternately. In practice, however, there is wide variation in arrangement and method of operation.

Filling—Soda-ash Solution.—Soap is hardened and the detergency and durability increased by the addition of varying proportions of solutions of soda-ash and sodium silicate. The use of soda-ash solution requires its preparation previous to the date of its use so that it may cool and deposit impurities. The method of preparation depends upon the quantity used. For a small boiling or used only in small amount it may be made by dissolving dry ash in a small tank adjacent to the crutchers and provided with a live-steam coil. When used in large amount, a large steam-heated tank is required and preferably provided with a mechanical agitator, whereby manual labor is reduced and the solution of the dry ash facilitated. With soap too cool it is desirable that the soda-ash solution be hotter than usual; with soap too hot the contrary condition is desired. A soda-ash solution testing by the hydrometer 34° Bé. hot will be saturated, 36° Bé., on cooling. The percentage of sodium carbonate in soda ash solutions of different densities is shown in the table on p. 352.

Sodium silicate is used at a density of 40° Bé., in which condition it is received at the factory. The satisfactory incorporation of solutions of soda-ash and sodium silicate depends upon their amount and the condition of the soap. With soap too hot or too cold, or too soft either from soft stock or the accidental mixing of more or less nigre, or with insufficient mixing of filling, the appearance of the resulting product will be effected injuriously by the use of a given amount of filling. By the use of an excessive amount of filling the soap will tend to open and a smooth, close appearance cannot be obtained without the use of a binding or closing agent as so-called mineral soap-stock (a petroleum distillate intermediate in consistency between heavy cylinder oil and vaseline). Soda-ash solution hardens the soap immediately on mixing; silicate of soda on mixing tends to soften the soap, its hardening effect manifesting itself on the aging of the product. Addition of both at a lower temperature than the soap cools the soap according to the difference in the temperature and permits it to be framed with greater safety than if the filling were as hot or hotter than the soap. For settled rosined soap the addition of filling should not exceed 75 pounds of 36° Bé. soda-ash solution and 25 pounds of sodium silicate per frame. Addition of a greater amount is reprehensible. In cheap rosined soap a common addition comprises upwards of 150 pounds of 36° soda-ash solution, 50 pounds of sodium silicate, and 50 pounds of mineral soap-stock.

Operating Crutcher.—The soap is run from the box or tank, supplying a pair or series of crutchers, and which box in turn is fed continuously from the kettle. The addition of the first charge to a cold crutcher may cool the soap to a temperature unsuitable for best results, whereupon applying steam to the jacket of the crutcher is necessary. With the crutcher once heated and the soap at the proper temperature additional heat is unnecessary. When one crutcher is filled the soap is turned into the other, if the crutchers are operated in pairs, and soda-ash solution added to the first and well mixed, whereupon the silicate is added and the mass crutched until a portion removed on a trowel is smooth, glossy, tenacious, and

TABLE XXIII.—SPECIFIC GRAVITY OF SOLUTIONS OF SODIUM CARBONATE AT 15° C. (59° F.).

Spec. Grav.	Deg. Bé.	Deg Twad.	Per Cent by Weight		1 Liter Contains, Grams.		1 Gal. (231 Cubic In.) Contains, Av Ozs.	
			Na_2CO_3.	$Na_2CO_3 + 10H_2O$.	Na_2CO_3.	$Na_2CO_3 + 10H_2O$.	Na_2CO_3.	$Na_2CO_3 + 10H_2O$.
1.007	1	1.4	0.67	1.807	6.8	18.2	9.07	24.28
1.014	2	2.8	1.33	3.587	13.5	36.4	18.01	48.57
1.022	3	4.4	2.09	5.637	21.4	57.6	28.55	76.85
1.029	4	5.8	2.76	7.444	28.4	76.6	37.89	102.20
1.036	5	7.2	3.43	9.251	35.5	95.8	47.37	127.82
1.045	6	9.0	4.29	11.570	44.8	120.9	59.77	161.31
1.052	7	10.4	4.94	13.323	52.0	140.2	69.38	187.06
1.060	8	12.0	5.71	15.400	60.5	163.2	80.72	217.75
1.067	9	13.4	6.37	17.180	68.0	183.3	90.73	244.57
1.075	10	15.0	7.12	19.203	76.5	206.4	102.07	275.39
1.083	11	16.6	7.88	21.252	85.3	230.2	113.81	307.14
1.091	12	18.2	8.62	23.248	91.0	253.6	125.42	338.36
1.100	13	20.0	9.43	25.432	103.7	279.8	138.36	372.12
1.108	14	21.6	10.19	27.482	112.9	304.5	150.64	406.28
1.116	15	23.2	10.95	29.532	122.2	329.6	163.04	439.78
1.125	16	25.0	11.81	31.851	132.9	358.3	177.32	478.06
1.134	17	26.8	12.61	34.009	143.0	385.7	190.80	511.88
1.142	18	28.4	13.46	35.403	150.3	405.3	200.51	540.77
1.152	19	30.4	14.24	38.405	164.1	442.4	218.95	590.27

AT 30° C. (86° F.).

Spec. Grav.	Deg. Bé.	Deg Twad.	Per Cent by Weight		1 Liter Contains, Grams.		1 Gal. (231 Cubic In.) Contains, Av Ozs.	
			Na_2CO_3.	$Na_2CO_3 + 10H_2O$.	Na_2CO_3.	$Na_2CO_3 + 10H_2O$.	Na_2CO_3.	$Na_2CO_3 + 10H_2O$.
1.142	18	28.4	13.79	37.21	157.5	425.0	21.01	56.70
1.152	19	30.4	14.64	39.51	168.7	455.2	22.51	60.73
1.162	20	32.4	15.49	41.79	180.0	485.7	24.02	64.80
1.171	21	34.2	16.27	43.89	190.5	514.0	25.42	68.58
1.180	22	36.0	17.04	45.97	201.1	542.6	26.83	72.40
1.190	23	38.0	17.90	48.31	214.0	577.5	28.55	77.05
1.200	24	40.0	18.76	50.62	225.1	607.1	30.03	81.04
1.210	25	42.0	19.61	52.91	237.3	640.3	31.66	85.43
1.220	26	44.0	20.47	55.29	249.7	673.8	33.32	89.90
1.231	27	46.2	21.12	57.80	263.7	711.5	35.18	94.93
1.241	28	48.2	22.29	60.15	276.6	746.3	36.90	99.57
1.252	29	50.4	23.25	62.73	291.1	785.4	38.84	104.79
1.263	30	52.6	24.18	65.24	305.4	824.1	40.75	109.95
1.274	31	54.8	25.11	67.76	319.9	863.2	42.68	115.17
1.285	32	57.0	26.04	70.28	334.6	902.8	44.64	120.46
1.297	33	59.4	27.06	73.02	351.0	947.1	46.83	126.37
1.308	34	61.6	27.97	75.48	365.9	987.4	48.81	131.74

shows a rounded, not flat, surface in the crutcher. Should the soap be slow in joining, steam may be lightly applied with good results and the soap crutched to a suitable temperature for framing. With rosined soap made and settled as described, no trouble need be experienced on crutching. The condition of the soap as it flows from the kettle should be observed. It should not be open, otherwise filling cannot be well incorporated, although it may appear so with soap in the crutcher. On cooling in the frame, and later on cutting and drying, efflorescence and soda-cracks will invariably occur, especially when the soap is framed too hot and cooled slowly, a common mistake with soft-bodied stock. Crutchers of the Strunz type should be filled above the mixer-arms; crutchers with a vertical screw enclosed by a cylinder should be filled above the level of the latter, otherwise in both cases more or less air will be incorporated, making the soap soft and spongy. The rapidity of crutching depends upon the rate of filling the crutcher, the temperature of the soap, the amount of filling added, and the ease of its incorporation, requiring ordinarily from ten to thirty minutes for each charge. In removing the last frames from the kettle, dipping often being necessary, care should be taken to avoid an addition of nigre, which, if present in framed soap in a considerable amount, makes its return to a subsequent boiling necessary. The number of frames of soap obtained from a given kettle charge should be uniform, but varies more or less with the volume of the nigre, which is determined as stated by the character of the finish on the settling change.

Framing.—The smooth, glossy soap in the crutcher is discharged into the frame run beneath it, which when filled and the warm soap heaped lightly along a medial line is replaced by another. The length of time required for crutching and framing depends upon the size of the boil and the number of crutchers. With two crutchers discharging alternately a carefully and well-crutched soap, 50 frames may be filled in a day.

Yield of Soap.—In the accompanying table is shown the yield of anhydrous soap from the characteristic glycerides of stock commonly used in soap-manufacture. A typical analysis of fresh, cold, unfilled soap from mixed stearin and olein stock is as follows:

	Per Cent.
Fat anhydrides	61.80
Combined alkali, Na_2O	7.20
Soap	69.00
Water	31.00

Analyses of this character will not only depend upon the stock from which the soap is made but upon the coarseness with which the soap was finished on the settling change. However, in water content they will not vary greatly from a percentage equivalent to 50 per cent of the proportion of anhydrous soap. This is the basis of the soap-maker's common calculation of 50 per cent yield of soap from the raw material. With stock used in practice the yield varies as indicated by the theoretical figures shown in the accompanying table.

Glyceride and Chief Sources	Molecular Weight.	Yield on Saponification with Steam Under Pressure.		
		Per Cent Fatty Acids.	Per Cent Glycerol.	Per Cent Water Absorbed.
Laurin, cocoanut and palm-kernel oils	638	94.04	14.42	8 46
Myristin, palm-kernel oils	722	94 47	12.70	7.17
Palmitin, palm-oil and tallow	806	95.28	11.41	6.69
Linolein, linseed-oil	878	95.68	10.48	6 16
Olein, olive- and cotton-oils	884	95.70	10.40	6.10
Stearin, tallow and lard	890	95.73	10 34	6.07
Recinolein, castor-oil	932	95.92	9 88	5.80
Arachidin, peanut-oil	974	96.10	9.44	5.54

Glyceride and Chief Sources.	Molecular Weight.	Yield on Saponification with Caustic Soda.		
		Per Cent Soap.	Per Cent Glycerol.	Per Cent Caustic Soda Absorbed.
Laurin, cocoanut and palm-kernel oils	638	104.38	14.42	18.80
Myristin, palm-kernel oils	722	103.87	12.70	16.57
Palmitin, palm-oil and tallow	806	103.47	11.41	14.88
Linolein, linseed-oil	878	103.19	10.48	13.67
Olein, olive and cotton-oils	884	103.16	10 40	13.56
Stearin, tallow and lard	890	103.14	10.34	13.48
Recinolein, castor-oil	932	103.00	9.88	12.88
Arachidin, peanut-oil	974	102 87	9.44	12.31

The reason for the greater yield of soap from cocoanut-oil than from the ordinary animal and vegetable fats is thus made clear. Not only is there a greater yield of anhydrous soap in itself, but in virtue of that higher yield from stock characterized by a high percentage of fatty acids of low molecular weight a greater degree of hydration in the finished product is permissible.

The yield of soap in frames is of course affected by the amount of filling used, especially to be noted in case of soap made by the cold and semi-boiled processes. The yield of soap from a given amount of stock and rosin, and from an unrosined stock as well, killed in a clean kettle, may be estimated as follows: Multiply the number of frames of soap by the weight of soap minus the filling in each; to this product add the weight of soap in the nigre. The weight of soap of the same degree of hydration as good soap in the nigre may be closely approximated by calculating the volume of ungrained nigre left in the kettle and considering its weight equivalent to that of water. From an analysis of a typical nigre previously given we may consider it to contain 30 per cent of anhydrous soap. Add to this calculated weight of anhydrous soap 50 per cent of its weight to allow for water. The result gives roughly the weight of soap in the nigre corresponding in hydration to that of good soap. This added to the weight of soap minus filling in the frames gives the total yield of soap from the stock used.

CHAPTER X.

MILLED-SOAP BASE. FLOATING SOAP. SHAVING-SOAP. MEDICATED SOAP.

Milled-soap Base: Stock. Stock Change. Strengthening Change Pickle Change. Settling Change. Framing. Floating Soap: Stock. Procedure. Crutching. Specific Gravity. Drying. Nigre. Waste Lye. Shaving-soap. Medicated Soap. The True Function of Soap as a Medicament. The Disenfectant Power of Common Soaps.

Milled-soap Base.—A toilet-soap is one intended for personal ablution and may be made by any process. The finest quality of toilet-soap is made by so mechanically treating dried, grained soap which has been reduced to chips and further dried that its beauty and durability have been increased without a sacrifice of detergency. A milled soap is a dried, grained soap reduced to laminæ and made compact by pressure. Such a soap may be greatly improved by the application of perfume and color.

Soap intended to be milled may be made by either the cold or semi-boiled process, but for the best results, viz., perfect saponification, neutrality, and purity, the grained, settled process is best.

Stock.—The influence of the varying admixture of different fats and oils upon the physical appearance and detergent properties of soap made from them has been already described. Only the best stock is available for milled soap base. The development of the art of perfumery and the use of colors have given the manufacturer of milled soap greater latitude in the selection of stock; nevertheless the best results in perfumery and coloring of milled soap can be obtained only with the freshest and purest material, which must undergo complete saponification by thorough boiling and further purification by settling.

Without the repetition of the character of stock ingredients it may be stated as a general rule that stock suitable for floating soap makes a first-class base for milled soap, and any depreciation of quality from this standard may be made according to the selling price and color of the finished milled goods. Rosin of WW grade is a desirable ingredient up to 10 per cent of tallow used. A good combination of ingredients is secured in 10 to 25 per cent of Cochin cocoanut-oil and 75 to 90 per cent of tallow of edible grade. The solubility of soap made of this combination may be increased by the addition of 10–15 per cent of oleo or cottonseed-oils for an equivalent weight of tallow. Rosin may be used for this purpose in colored soap. The mildness of soaps made from palm- and olive-oils adapt them for use in milled soap. The characteristic stock does not depart greatly from cocoanut-oil and tallow combined with softer stock in the proportions previously mentioned. Assuming a stock charge of 75 per cent prime tallow and 25 per cent cocoanut-oil, the procedure of milled-soap base manufacture is substantially as follows.

Stock Change.—Tallow of first-class color, odor, and body is run into the kettle and simultaneously lye at not over 15° Bé. Emulsion ensues promptly on boiling. The lye during saponification should be present in excess but not sufficient to grain the soap. Every effort should be made to secure thorough saponification, for upon this factor rests the success of the process. If the soap be grained during the stock change saponification is retarded by the soap being thrown out of solution and enclosing in the grain more or less free fat. Should the supply of lye in the kettle be deficient the mass may stiffen, which condition should be carefully guarded against, as it interferes greatly with thoroughness of saponification. Towards the close of the change, as absorption proceeds more slowly, add the lye at 18° Bé: and in successive additions of small amounts on prolonged boiling; before graining use it at 20° Bé. The procedure at no stage should be hurried, for thereby thoroughness of saponification may be sacrificed. On the complete absence of a greasy feel to the soap as it is cooled and pressed between the fingers, with the tendency to be reduced readily to chips or flakes, the tallow soap may be grained. This may be done with either salt or lye at 30°–40° Bé., most conveniently with the latter. The soap is reduced to a fine grain and the strong stock lye withdrawn for utilization in the boiling of rosined soap. The cocoanut-oil is now added to the grained tallow soap and killed with lye at 20° Bé., gradually increasing the density to 25° Bé. as saponification proceeds to the close. In killing the tallow first and the cocoanut-oil afterwards, the capacity of a given kettle can be fully approximated, as the combined swelling of the tallow and cocoanut-oil would considerably reduce the amount of stock that could be killed at once in a kettle of a given size. With ample kettle space, equal thoroughness of saponification, it is claimed, may be effected by killing the tallow and oil together with lye first at 18° Bé. and increasing it to 25° Bé. towards the close. However, wherever practicable, stock should receive lye at the density most suitable for thorough combination, which condition is obtained in the procedure first outlined. The tendency of cocoanut-oil to swell and rise rapidly in the kettle to the extent of boiling over should be carefully watched and may be checked by the addition of strong lye, salt or pickle or cold water, in which event, of course, steam for the time should be turned off.

The peculiar taste of cocoanut-oil suggestive of strength should not be confused with the taste of genuine strength, otherwise more or less oil will pass the stock change unsaponified. The boiling should be prolonged until assured of complete saponification, when the soap may be grained. This may be done, as on the tallow change, with either strong lye or salt. Whether the latter is dry or in solution will depend upon conditions in the kettle. With a large amount of water in excess, dry salt should be used. If rosin be an ingredient of the milled-soap base, it is added after the withdrawal of the final stock lye and killed with 20° Bé. lye under the same conditions and by the same procedure as followed on the rosin change of a settled rosined soap, after which the soap is ready to strengthen.

Strengthening Change.—The soap may be strengthened, after withdrawing the rosin lye, if rosin be used, either by adding sufficient water, boiling meanwhile, to just close the soap, and then adding 20° Bé. lye until open and continuing boiling throughout the period allotted to this change. The absorption of alkali should be carefully observed, as it will serve as an index of the thoroughness of the saponification on the stock change. As absorption takes place as indicated by the reduction of strength in the lye, and also by the soap tending to close, more lye at 20° Bé. should be added. A great excess of strong lye on the strengthening change is not essential; its addition from time to time to replace the absorption and to maintain the soap in a flat grain suffices. Towards the end of the change with strength present, but with the soap tending to stiffen and thereby to interfere with even, regular boiling, either water or pickle should be added according to conditions. With the soap well open and tending to boil explosively from stiffening, water may be added to dilute the graining strength present, but when tending to close, with sufficient strength present for absorption but insufficient for keeping the soap open, pickle should be used, its density up to 25° Bé. depending upon the amount of water in the kettle. The completion of alkali absorption is indicated by the intensity of strength in the lye remaining permanent. After boiling for some time under these conditions steam is turned off and the strength lye allowed to settle out. After withdrawing the strength lye more or less caustic alkali will be mechanically retained by the grained soap, which to make the soap neutral it is necessary to remove by washing with pickle. This wash or pickle change may have to be repeated as often as three times in order to remove the last traces of strength and to produce a perfectly neutral soap ready for settling.

Pickle Change.—The strength mechanically retained by the grained soap left on withdrawing the strength lye is best separated by closing the soap by the addition of water boiling meanwhile. No more water should be added than is sufficient to flatten the grain to the point of joining. Pickle at 15°-25° Bé. is then added and thoroughly boiled through. The result is that the soap is again grained, and in the water withdrawn from the soap and reduced to salt solution by the pickle added, is the strength. After having worked the pickle well through the soap the pickle lye is settled out. Pickle at 10° Bé. may be added to the soap after withdrawing the strength lye, and well boiled through without the previous addition of water to close the soap. When the latter procedure is followed the density of pickle required to again grain the soap will depend upon the degree of hydration of the soap. With addition of considerable water and the soap closed, 25° Bé. pickle will be required; with a less degree of hydration of the soap the density of the pickle is reduced. After withdrawing the pickle lye a comparison of the strength therein with that remaining in the soap will determine the efficiency of the wash and the amount of strength remaining in the soap. With an excessive amount of strength on the strengthening change, the amount mechanically retained will not be removed by one wash, and the degree of completeness with which it is to be removed will be determined by the grade of milled soap made from it.

With the soap neutral, or the strength reduced to satisfactory proportions, the soap is ready to settle.

Settling Change.—The purpose of settling is to reduce the soap to a well-defined consistency by means of water, whereby it is in a condition to settle out the impurities on standing and cooling. During this period the strength is also settled out, the degree of thoroughness of separation depending upon the length of the period. Examination of soap in successive layers as framed indicates an increase of free alkalinity as the nigre is approached where it reaches the maximum. The soap is settled as follows: Water is carefully added to the kettle and well boiled through after each addition, pains being taken that no excess be used, during which time the soap is closed, rises in the kettle, and from the incorporation of water and steam assumes on the surface a stiff consistency which, as the incorporation of water proceeds, boils up evenly and rolls over, the surface breaking into numerous placques or saucer-like formations. It should be noted particularly in this connection that with milled-soap base, floating soap, and all unfilled soap the addition of water beyond that supplied by condensation of steam from the open steam-coil as a rule is unnecessary to effect the degree of hydration desired where ample time is allowed for the settling change. Soap hardened in the crutcher by the addition of soda-ash solution need not be finished so coarse as is required for soap of the kinds mentioned. The purpose of settling, as stated, is to finish the soap in such a condition that a nigre containing the impurities will form on standing and cooling. In this there are two extremes, viz., the soap may be finished so thin by the addition of an excessive amount of water that no nigre separates; it remains distributed throughout the mass of soap. In other words, a nigre does form, but it includes practically the entire mass of soap. The other extreme obtains with the addition of so little water that the soap is not completely joined, with the result that a weak waste lye separates. With the latter condition in mind, the aim on settling milled-soap base, floating, and all unfilled soap is to incorporate, either by the direct addition of water or by the condensation of steam from the live-steam coil, just sufficient water to thoroughly join the soap, to make it homogeneous, and to carry it past the joined state only so far as to introduce conditions conducive to the formation of the minimum amount of nigre and the maximum amount of good soap. With the kinds of soap mentioned and with ample time for the change, these conditions are best introduced by prolonged boiling with a live steam coil. Conditions affecting the formation of nigre are gone into in the discussion of settled rosined soap. When the physical conditions previously described appear on the surface of the soap and are permanent, steam is turned off securely and the soap allowed to cool. With small batches not exceeding 10 15 frames, made in a small kettle, the kettle should be covered with suitable material to avoid too rapid cooling.

Framing.- With a coarse finish as described nigre forms readily and at a rate depending upon the mass of soap and the season. For any particular kettle the number of days to allow for cooling is determined by experience. For a small kettle in an exposed position yielding a dozen frames, one day intervening between settling and framing may suffice. It is not necessary to observe the same temperature limitations of filled, settled, rosined soap. The soap should not be drawn off at a temperature higher than 160° 170° Fahr., as the formation of nigre may not be complete and as unnecessary labor is required for cooling in the frame. Soap made from stock used for milled-soap base cools quickly, and unless framed at the right time the yield may be reduced by the soap cooling and adhering to the sides of the kettle. The soap may either be run directly to frames and there hand-crutched until stiff, or it may be run first to the crutcher, where, if cooling by means of a water-jacke is available, the labor of hand-crutching in the frame may be reduced.

Floating Soap.—The procedure to be followed in floating-soap manufacture up to the crutching stage is substantially as already outlined in the manufacture of milled-soap base, which procedure is also applicable to the manufacture of ordinary white settled soap, of which little, if any, is now made, and to the manufacture of genuine castile soap from olive-oil or olive-oil foots and their common substitutes, viz., cottonseed-oil and tallow. The manufacture of floating soap is of comparatively recent origin in the United States, having been carried on only for the last twenty-five years. Floating soap is claimed to be of Chinese origin and to have been first imported into England in the middle of the eighteenth century. This soap was made exclusively from cocoanut-oil. After its original manufacture in the United States it soon acquired great popularity as a good, cheap toilet-soap and its manufacture became general. Its floating property, due to the reduced specific gravity from the incorporation of air by special crutching, indicates the absence of all inert or inferior detergent matter and its freedom from color, the use only of the best raw material. Floating soap is applicable for all detergent purposes, but its wastefulness in use restricts its application to toilet purposes and to the more delicate cleansing. Floating soap should be free from color or of a shade no deeper than that of cream, with no odor arising from rancidity of cocoanut-oil, and, according to custom, should not be highly perfumed. Saponification should be complete, with no traces of uncombined stock, otherwise a rancid odor develops in use. It should lather freely, but not excessively, with thick, firm lather. The pressed bar should not warp on aging or show signs of local discoloration, and the general discoloration peculiar to all white tallow soaps on aging should be very slight. Its specific gravity should be less than that of water, otherwise the soap becomes a white settled soap with no buoyancy.

Stock.—Cocoanut-oil, commonly of Ceylon grade, although bleached copra-oil is also used, is an indispensable ingredient.

It imparts fluidity to the firmer tallow soap when hot. It imparts quick and profuse lathering properties and aids brightness of color and tends to preserve it. It is very important that the oil should be completely saponified, otherwise rancidity develops in use, as shown by the odor of the fresh surface and of the hands after washing.

The tallow should be of high grade and, if used unbleached, should approach edible stock in quality. It forms the basis of the soap. By its admixture with the more soluble cocoanut-oil soap forming a thin, quick lather, the firm, slow-forming lather of tallow is softened and developed more quickly. If it be desired to obtain the blandness or mildness in use peculiar to olive-oil soap, well-refined cottonseed-oil or oleo-oil may be substituted for a portion of the tallow or a softer tallow base used.

All tallow soaps discolor more or less on aging, whether carefully preserved as samples or exposed upon the market, according to the proportion and quality of the tallow and the nature and source of any olein stock if used. Cocoanut-oil is commonly used in proportions varying from 10 to 30 per cent, it being generally believed that 20 to 25 per cent is the minimum to produce the best results; the remainder of the stock may be varied according to suggestions previously made.

Procedure.—The procedure of boiling settled tallow-cocoanut-oil soap has been already described in the manufacture of milled-soap base, and attention at this place may be directed only to its general consideration. The prime essentials are thorough saponification, neutrality on the settling change, and a coarse finish. Without thorough saponification rancidity soon develops in the use of the product; with free alkali present the excoriating effect of the soap unfits it for toilet use; and unless the soap is well boiled and finished coarse a large nigre is formed, whereby the yield of good soap is reduced and the efficiency of manufacture sacrificed. The time required for manufacture comprises, as a rule, four days, although this depends upon the size of the boiling, with the following stages of treatment: The stock change, which may require two days for its completion when the tallow and oil are killed in separate charges, the strengthening change, which requires one day; the pickle or wash change to remove excess of free alkali, which requires a period determined by the ease and degree of thoroughness with which the excess of alkali may be removed; and the settling change, which requires a period of time depending on the size of the boil and which is completed in one day. Floating-soap scrap should be added to the kettle on the strengthening change. The separation of free alkali is always a troublesome matter and may be effected as described by one or more pickle changes or by neutralizing the alkali in the crutcher, or by both methods. As to whether the waste lyes are withdrawn neutral or with more or less free alkali and treated subsequently with fresh stock for its recovery before evaporating the lye for glycerin will depend largely upon the amount of soap of this kind made. It is only after considerable experience that each change can be made complete and the waste lye from each change withdrawn neutral where neutrality is desired. By leaving more or less stock unkilled on each change preceding the strengthening change, the waste lye may be withdrawn with a minimum percentage of caustic soda. More care and time are then required on strengthening change to complete the saponification purposely left incomplete on the preceding changes.

The period required for cooling and deposition of the nigre depends upon the size of the boil, which influences the rate of cooling. As a general rule heavy-bodied unfilled soap requires the shortest time, which increases with the softness of the body and size of the batch and is influenced by the season and exposure of the kettle to influences retarding or accelerating the loss of heat by radiation.

Crutching.—The high solidifying point of floating-soap stock requires its withdrawal from the kettle at a much higher temperature than soap made from softer stock, as rosined soap. The soap should not be withdrawn at less than 170°–180° Fahr. The procedure followed in crutching will be determined by the volume of soap made. With small batches from which free alkali may be readily separated by washing with pickle, crutching comprises the addition of soap to the crutcher at about 175° Fahr. up to a certain level in the crutcher according to its design and as determined by experience. Upon completion of crutching the soap will have cooled to about 130° Fahr., and from its expansion due to incorporation of air will have completely filled the crutcher. With crutchers having vertical agitators the maximum level of clear hot soap before reversing the driving-belt may be indicated either on the vertical shaft or inner concentric cylinder enclosing the screw. With crutchers of the Strunz type, the level is indicated on the vertical blades of the horizontal shaft. Incorporation of air is effected by simply reversing the driving-belt, whereby conditions are introduced exactly contrary to those prevailing when filling is incorporated. As agitation proceeds the soap loses its clearness and becomes opaque, the desired specific gravity being determined by either allowing the crutcher to completely fill or by testing the buoyancy of a small sample of soap in water. Should it be desired to hasten cooling, water may be introduced into the jacket of the crutcher. If the proper buoyancy is obtained before the soap is cool enough and it is then framed, the buoyancy of the soap tends to be diminished by its coalescence in the frame, whereby the lower portion of the frame is of higher specific gravity than the upper portion. Satisfactory air incorporation is determined by the opacity of the soap and its volume in the crutcher and its tendency to solidify at the edges or circumference of the crutcher, although these indices may be supplemented by use of the thermometer and the floating-test. Just previous to framing perfume is added. The time required for crutching depends upon the initial temperature of the soap, the use of the cooling-jacket or the season, and the proportion of soap to be crutched at each charge to the capacity of the crutcher. The rate of crutching with the regular soap-crutchers does not exceed that for filled soap. The time required need not exceed 15 to 20 minutes.

In the frame the soap cools quickly and sinks along a medial line, leaving on the sides more or less soap, which on cutting forms scrap. This proportion may be reduced by depressing it as it forms into the mass of softer soap.

With floating soap made in large batches, the removal of free alkali as stated is a more troublesome matter than with small batches. Recourse is had to neutralizing the remaining free alkali in the crutcher. As previously stated, the proportion of free alkali increases as the nigre is reached where it attains its maximum percentage. On large-scale production the good soap may be pumped to a long tank ample in capacity for a single boil surmounting a series of crutchers and containing an agitator. Here the soap may be mixed to a uniform consistency and the free alkali determined once for the entire boil. The percentage of free alkali having been determined, the quantity of material calculated per frame for its neutralization may be added to the charge of each crutcher. The neutralizing agent may consist of cocoanut-oil, which, however, is not always to be recommended; boric acid, which, in combination with free alkali, forms sodium biborate, or borax; melted stearic or oleic acid, the product of combination with which being soap. The neutralizing agent is added at the beginning of the crutching, the efficiency of neutralization being determined by testing a freshly cut surface of a sample of crutched soap with an alcoholic solution of phenolphthalein. On application of a drop of this indicator the presence of free alkali is shown by a pink coloration. With the desired specific gravity attained the soap is framed as described.

Specific Gravity.—Floating soap is merely well-made settled soap diluted with air to the degree that it is lighter in weight than an equal volume of water. The specific gravity is a very variable factor not only in soap of the same stock composition but in different frames of the same boil and in different parts of the same frame, and in the same soap cut and with different degrees of dryness. The specific gravity of any soap depends upon the stock used and the degree of hydration and filling for a soap heavier than water; for a floating soap it depends upon the proportions of air and soap in a given volume and upon the dryness or degree of hydration. The following analyses and determinations of density of a floating soap in successive stages of manufacture are interesting as showing the change in composition and density ensuing on the incorporation of air.

Drying.—Supplementary to the general principles of soap-drying already discussed it may be stated that the soap should be allowed to stand as long as possible in the frame before cutting, and to prevent warping or uneven drying, and to reduce the tendency to discoloration, it should be allowed to dry more slowly and at a lower temperature than filled soap. To counteract wastefulness in use peculiar to all soap filled with air and containing cocoanut-oil, the drying process should be more complete than with ordi-

Constituents of the Soap.		Soap from Top of Kettle Before Crutching.	Same Soap After Crutching and Partly Neutralizing the Free Alkali.	Same Soap Near Nigre Before Crutching.
Soap	Fatty acids	61.20 per cent	63.30 per cent	58.20 per cent
	Combined soda, (Na_2O)	7.66 "	7.92 "	7.28 "
Free caustic soda, (Na_2O)		.08 "	.04 "	.42 "
Sulphate and chloride of sodium		.42 "	.43 "	.58 "
Water		30.64 "	28.31 "	33.52 "
Specific gravity		1.265	.922	1.272

nary soap. Analyses of floating soaps on the market indicate a moisture content varying from 11 to 22 per cent.

Nigre.—It is not advisable to incorporate the nigre of settled white soap into the succeeding boil, as the color of the finished product would thereby be greatly deteriorated. On the removal of the supernatant good soap to the crutcher, the nigre is boiled up and given a sharp grain with dry salt. The waste lye after settling out is discharged into the sewer. The nigre may be utilized by rosining it up to a given percentage and incorporating it into the best grade of rosined soap; or it may be transferred to a smaller kettle and settled, the product going, as desired, into "castile" soap, floating tar soap (it being necessary to add the neutralized pine tar in quantity sufficient to produce the color and odor desired to the soap in the crutcher), so-called disinfectant soap (made by introducing a small amount of disinfectant sufficient to produce the color and odor desired), or it may be used as toilet-soap base for the less expensive grades of milled goods. It is a "clean" nigre in comparison with those obtained from settled rosined soap and where a variety of detergents are made sufficient outlet for its utilization will be found. The proportion of nigre, likewise the yield of good soap, is determined by principles developed in the description of the manufacture of settled rosined soap.

Waste Lye.—Waste lye from white settled soap, such as is used for floating-soap and toilet-soap base, is of the highest degree of purity of all waste lye produced in the soap-works. As it comes from the best material it is richest in glycerin and, owing to the purity of the vehicle, it is recovered therefrom with the least trouble and expense. Wherever practicable stock lyes should be withdrawn neutral and discharged directly to storage-tanks. Here they are allowed to cool and to separate any soap held in solution, which on removal is returned to the nigre or rosined up in a boil of rosined soap. Strengthening lyes are worked over in separate kettles with fresh stock which on withdrawing the neutral lye remains as a basis for a new boiling of soap, either rosined or white, as desired. Pickle lyes are of small volume and may be used at once in rosined soaps on either the rosin or strengthening changes.

Shaving-soap.—The manufacture of shaving-soap is a specialty confined to but very few concerns with whom the article has attained a high degree of perfection. Shaving-soap should yield a profuse, firm, and permanent lather; the soap should not discolor or harden on aging or develop a rancid odor. The soap-stock required is tallow of edible grade. In spite of numerous formulæ that are continually being published, to secure the best results cocoanut-oil or cottonseed-oil is inadmissible as a stock ingredient and likewise directions for manufacture by the cold and semi-boiled processes are equally unreliable. With regard to saponification, it must be perfect and the product must be a soap of absolute neutrality. These essentials require the use of the grained process of manufacture. The care required necessitates the manipulation of comparatively small batches, the general procedure of boiling having been outlined already in the description of boiling milled-soap base and floating soap. The essential requisites, that the soap shall not harden on aging and shall yield at once a profuse and lasting lather, require the use of a proportion of caustic potash; analyses of a popular brand of shaving-soap show that of the combined alkali in the finished product 25 per cent is potassium oxide and 75 per cent is sodium oxide, thus indicating the relative proportions of caustic potash and caustic soda used. Analyses also indicate a very low moisture content, without which the soap would dry, warp, and tend to discolor on aging. To secure the qualities requisite shaving-soap must be milled. A plodder of the type generally used in the milling process is shown in Fig. 158. The satisfactory incorporation of glycerin to which the emollient and non-drying properties of the soap are largely due is possible only by milling. Analyses indicate the presence of 8 to 10 per cent of glycerin. The firmness and permanence of the lather are aided by the incorporation of a gum, usually tragacanth, which being soluble in water affects the lather at once. Gum is present in the finished soap to the extent of about 1 per cent. The incorporation of glycerin, gum, and perfume, if any, is effected during milling.

Medicated Soap.—The manufacture of soap possessing special properties in virtue of the addition of substances of which these properties are characteristic—as a rule medicinal agents and disinfectants—is a branch of the soap industry of some magnitude. The soap-base which serves as the vehicle for the medicament may be made by either the cold, semi-boiled, or grained process. The absence of free alkali being an essential requirement of the finished product, together with a fair degree of durability in use, so-called milled soap, which is prepared usually by the grained process, is the most common form of detergent used for the incorporation of such bodies as commend themselves for medication by the skin.

The True Function of Soap as a Medicament.—As the curative property of the so-called medicated soap is reputed to reside in the medicament added, it may be well to examine the character of "medicated" soap in particular and soap in general in this respect. The ease with which medicinal agents can be incorporated with soap and their intimate, though brief, and assumed remedial contact with the affected surface during ablution give rise to this class of toilet-soap, especially the milled variety.

Medicated soap may be divided for our present purpose into two general classes, viz., prophylactic and disinfectant soaps, according to the character of the substance added to the soap. By a prophylactic is understood any agent that will prevent disease; by a disinfectant, any agent that will kill pathogenic or disease-producing bacteria. Antiseptics differ from disinfectants only in degree. An antiseptic will retard bacterial growth; a disinfectant will entirely prohibit it. Disinfectants in solutions too dilute for effective germicidal action, or applied to the affected surface in any manner whereby their full efficiency is diminished, become antiseptics and to a more extreme degree worthless for their intended purpose.

To that soap of the character which we are discussing the terms "medicinal" and "medicated" are loosely applied. We shall see before concluding that all soap, irrespective of quality or purpose, is medicinal, i.e., prophylactic, while only that to which a specific medicament is added is medicated.

Before proceeding further, it will be well to consider the nature of these rudimentary parasitic organisms called bacteria.

Bacteriology teaches that bacteria are the smallest of living things yet known. They are not animals, but are members of the vegetable kingdom and are possessed of definite yet varying shapes. They consist of a jelly-like substance called protoplasm which is covered in and held in place by a well-formed membrane of a relatively hard and dense character, exactly similar in composition to the woody fibre of trees.

According to their shape the bacteria are divided into three chief groups, called respectively cocci, bacilli, and spirilla. The cocci are spherical bodies and may exist singly or in pairs, in fours, in clusters, or in chains. In this group we find the smallest bacteria known, many of them not over $\frac{1}{150000}$ of an inch in diameter. The bacilli are rod-like bodies, varying much in size in different species and in members of the same species. They are larger than the cocci, measuring in length from $\frac{1}{25000}$ of an inch to $\frac{1}{4000}$, and in breadth from $\frac{1}{125000}$ to $\frac{1}{16000}$ of an inch. Many varieties are possessed of organs of locomotion called flagella.

The spirilla resemble the bacilli except that they are twisted into corkscrew shapes or have gently undulating outlines. Upon an average they are much longer than the bacilli, one species being very long, measuring about $\frac{1}{600}$ of an inch. As seen in the natural state bacteria are found to be colorless, but it is by the application of various aniline dyes that they are usually studied. These minute plants increase by a simple method of division into two equal parts, or by a more complex process of forming a seed—the so-called spore—which later on develops into the adult form. Under favorable conditions they are able to multiply at an enormous rate; for instance, it has been calculated that a bacillus dividing one every hour would at the end of twenty-four hours have increased to 17,000,000; and if the division continued at the same rate we should find at the end of the third day an incalculable number of billions, whose weight would be nearly 7500 tons!

But fortunately for our welfare, Nature by various means renders the possibility of such a happening entirely beyond the slightest chance of realization, her greatest barrier being the lack of an adequate food-supply.

The distribution in nature of bacteria is well-nigh universal, occurring as they do in the air we breathe, the water and milk we drink, upon the exposed surfaces of man and animals and in their intestinal tracts, and in the soil to a depth of about 9 feet. But it has been noted that at very high altitudes and in glacier ice none exist, while in the Arctic regions and at sea far from land their numbers are very few.

The conditions governing their growth involve many complex problems, but a few of the chief factors concerned are moisture, air, food, temperature, and light. All bacteria must have moisture, else they die sooner or later, depending upon the hardiness of the species, and none can multiply without it. A supply of air is by no means essential to all germs. To some it is absolutely necessary, and such germs are called aerobes. To others air is wholly detrimental, and they constitute the anaerobes; while to the majority of bacteria air-supply is a matter of indifference, and in consequence they are grouped under the term facultative anaerobes.

The food supply of many consists of dead animal and vegetable materials, a few require living tissues, whilst a small number can exist wholly upon mineral salts or even the nitrogen of the air. The lowest temperature at which some bacteria can multiply is the freezing-point of water and the highest 107° Fahr. However, the average range of temperature suitable to the majority lies between 60° and 104° Fahr., $98\frac{3}{5}^\circ$ Fahr. being the most suitable for the growth of disease-producing germs. Light, ordinarily diffused daylight, or its absence, is a matter of no moment to most germs, whereas direct sunlight is a destroyer of all bacteria.

The application of soap in the treatment of germ disease is confined to its use as a prophylactic in removing pathogenic germs from the surface of the body and to its assumed use as a disinfectant in checking the progress of germ life that may have secured lodgment thereon. As a prophylactic the use of soap has been claimed to extend to the field of medication by the skin.

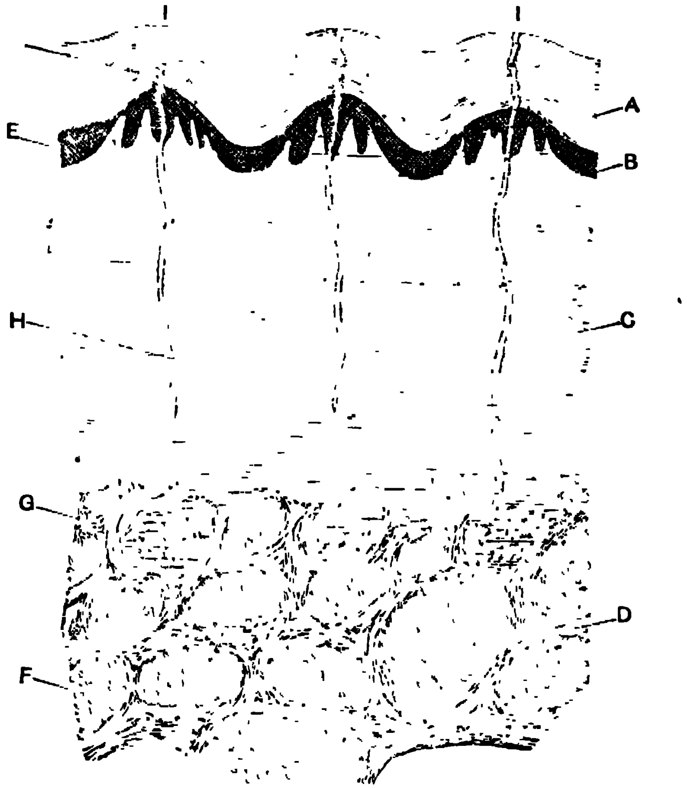

FIG. 141.—Vertical Section of the Skin.—*A*, epidermis, *B*, dermis, or cutis vera; *C* tissue beneath skin, *D*, connective tissue, *E*, tactile corpuscles; *F*, fat tissue; *G* sweat-glands; *H*, tubes of sweat-glands, *I*, orifices of sweat-glands.

In a discussion of the use of soap for this purpose, as a vehicle for the medicament, it will be necessary to consider the structure and function of the human integument.

The skin is composed of two layers, viz., the under layer, called the *dermis*, or *cutis vera* (true skin), containing nerves and blood-vessels, and an exterior layer, the *epidermis*, formed from the dermis and composed of flattened epithelial cells, which protect the sensitive true skin below, and which are continually being shed as they lose their vitality. It is on this portion of the integument that micro-organisms lodge.

The skin is primarily an organ of elimination, not of absorption. While the distribution of the total excreta of the human body is extremely variable, it may be divided approximately in the following way, although it must be remembered that the separate proportions vary greatly under variable circumstances; by respiration about 32 per cent; by secretion through and evaporation from the skin about 17 per cent; by the kidneys about 46 per cent, and by the intestines 5 to 9 per cent.

Medication by the skin contradicts a natural function and can be employed with satisfactory results only by the sole use of those vehicles, e.g., lanolin, etc., which possess marked penetrating power, and which must remain in contact with the affected surface for a considerable length of time. Pure lanolin is perfectly neutral, is very difficult to saponify, and has no tendency to become rancid; its capacity for absorbing water is remarkable, taking up when kneaded with water about 110 per cent, forming a plastic, cream-like fat; it also combines freely with glycerin; in both forms—hydrated or with glycerin—it forms an excellent basis for ointments, pomatums, and for cosmetic purposes. The extraordinary capacity with which lanolin and lanolin ointments are rapidly absorbed by the skin affords an unrivalled method of introducing effective remedies through the skin; as an evidence of how promptly this absorption and action takes place the fact may be mentioned that, for instance, a lanolin corrosive sublimate ointment containing but $\frac{1}{1000}$ per cent of sublimate produces the metallic taste upon the tongue within a few minutes after application on any part of the body, as also that upon the application of a 10 per cent potassium iodide lanolin ointment, the presence of iodine in the urine will make its appearance in about one-half to three-quarters of an hour after application. No one of the known fats approaches this extraordinary power of absorption either by the skin or of water; lard associates with but 15 per cent of water, and petrolatum stands in this respect lowest on the scale of all fats, absorbing but 4 per cent of water and being least absorbed by animal tissue. It will be evident that the efficiency of such agents is greatly diminished by their incorporation with bodies possessing little or no penetrating power, e.g., soap, and their results rendered nil by their application under the conditions associated with the use of soap in ablution.

Too little emphasis has been placed upon the properties of soap itself as a prophylactic, the tendency being to attribute the natural properties of the detergent to the substance incorporated with it, when, with the proportion commonly added and under the conditions prevailing in use, the application of the soap is utterly without results that can be justly attributed to the medicament or other agent added. For the remedial agent to be effective in use, it must be present in the soap in such proportions as would make the soap unsalable as a detergent through decomposition, efflorescence, repulsive odor, or generally unattractive appearance. Owing to the small quantity of the medicament commonly present, often its inertness or insolubility in water, its incompatibility with the soap resulting in decomposition' whereby whatever virtue it possessed is destroyed, and the unfavorable conditions affecting its use, the so-called medicated soap has little, if any, greater curative power than ordinary soap.

A pure, neutral soap lathering freely in cold water, e.g., an olive-oil soap, or any made from suitable stock admixture, as a floating soap, is at once the cheapest and most effective prophylactic known to preventive medicine.

Turning to medicated soaps containing disinfectants, other natural properties of soap are exhibited. The disinfectants commonly added to medicated soap of this character are mainly of coal-tar origin, consisting of phenol (carbolic acid) and its homologues.

Emphatically true is it of disinfectant soap that soap intended for any detergent purpose cannot be made a satisfactory vehicle for the odorous disinfectants in effective amount; that when added to the soap-base in amount seldom exceeding 10 per cent, the natural germicidal property of the soap itself is very little increased. This fact has been demonstrated by actual experiment * with 2 per cent solutions of pure curd and disinfectant soaps in contact with bacteria during periods varying from five minutes to four hours. Unless in prolonged contact with the surface of the skin, soap is without disinfectant power, and even though impregnated with a germicide, in the hasty process of personal ablution, with either quality of soap, its germicidal power is reduced to zero. With respect to their energy as germicides, soap solutions differ but little. They are all equally capable of being used as disinfectants in all cases

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where they can be employed to advantage. The advantage they possess over other disinfectants consists in their ready application and in their complete freedom from danger. As demonstrated by comparative experiments, they rank low where quickness and efficiency are desired.

The curative property generally attributed to medicated soap does not reside primarily in the medicament added, but is inherent in the soap itself and is shared by every soap. The remedial value of soap lies, therefore, in the mechanical action associated with its use as a cleansing agent.

The soap solution emulsifies and removes the oil exuded by the sweat-glands through the pores of the skin and the cast-off epithelial cells with the germ-laden dirt that has collected upon them.

The remedial action of all soap, medicated or otherwise, is essentially mechanical. It removes the dirt and germs and softens the skin, the friction of ablution exercising and stimulating the excretory glands and providing for the freer elimination of waste matter, the unobstructed discharge of which is essential to normal health.

With the epidermis thus cleansed and softened, the skin is in a suitable condition for complete disinfection, e.g., with corrosive sublimate (mercuric chloride) solution 1:1000, as practised by surgeons, or for the application of any medicament employed with best results alone or in a more suitable and effective medium than detergent soap.

The Disinfectant Power of Common Soaps.—A large number of chemists have asserted the disinfectant power of common soap, but they are not in agreement as to the necessary conditions to be fulfilled in its use. Professor Serafini, according to the *Revue d'Hygiene*, has tried to settle the matter by experiments which he directed at Padua. His conclusions are as follows:

"1. Both hard and soft soaps have a marked disinfectant action, due to the soap itself, and not to uncombined alkali or fatty acid.

"2. The alkalinity of a good soap is so slight, even in concentrated solution, being only .14 to .192 per cent in a 5 per cent solution, that it is quite insufficient to account for the disinfection.

"3. The alkali set free on dissolving the soap would be equally ineffective alone, although it no doubt helps the general result.

"4. As soap is not completely soluble in cold water, it is to the soluble part that the action must be attributed. The disinfectant power is not altered by filtering the cold solution.

"5. Hard-water or other soap precipitants lessen the disinfecting action, as does the presence of large amounts of carbonates.

"6. Heat assists the action, both by bringing more soap into solution and directly.

"7. Ordinary filling is non-disinfectant. Hence the disinfectant power of the soap is lessened by its presence and in proportion to the extent to which the soap is filled.

"8. Soaps containing rosin are less disinfectant than other soaps in direct proportion to the amount of rosin present.

"9. Too much stress must not be laid upon the disinfectant power of soap for clothing. Strong soap solutions penetrate with difficulty, especially when the meshes of the fabric are clogged with foreign bodies. These, too, are often just those which cause the clothing to need disinfection. The solubility of the dirt in the soap solution, too, is slight or nil."

It will be at once observed that No. 8 is simply a corollary of No. 7. The difficulty alluded to in No. 5 can be cancelled by the simple expedient of using a larger quantity of soap. Soft soaps generally contain more water, glycerin, and other bodies which are not soap, and are therefore inferior in disinfectant power to the hard soaps. Colored soaps, too, are generally filled more than white soaps, and are therefore less reliable. It may be regarded as certain that a good washing with soap is a very valuable disinfectant process when no better can be had, and the soap should be used at a high temperature and with as little water as practicable. Clothing, too, should be soaked for some hours in the hot soap solution as a preliminary process.

It has been shown repeatedly that the soaps sold as disinfectant soaps are not more effective than the ordinary kind. It often happens that the disinfectant added to the soap, and on which the sellers rely, reacts with the salts of the fatty acids, with the result that the disinfectant powers of both are cancelled.

Professor Serafini attributes the opinion of Reithoffer, Beyer, and Heyden, who deny the disinfectant power of soap, to those chemists having experimented with low-class goods.

CHAPTER XI.

ESSENTIAL OILS AND SOAP PERFUMERY.

Essential Oils. Chemical Classification. Natural Sources. Methods of Extraction: Mechanical; Maceration; Enfleurage; Indirect Extraction with Volatile Solvents; Direct Extraction with Volatile Solvents; Distillation. Concentrated Essential Oils. Substitutes for Essential Oils. Natural Musk, Civet, etc. Adulteration. Table of Common and Possible Adulterants of Essential Oils. Examination of Essential Oils. Blending of Perfumes. Soap Perfumery. Perfuming Milled Soap. Perfuming Floating Soap. Perfuming Laundry Soap. Use of Colors. Organic Colors. Inorganic Colors. Application of Colors.

Essential Oils.—Organic nature abounds with a large number of bodies possessing an agreeable odor, which, with the exception of musk and civet, are of vegetable origin. To them the generic term of "essential" or "volatile" oils is given, the former being traditional and having reference to the fact that the odoriferous body was supposed to constitute the "essence" of the plant; the term "volatile" is more definite, but yet not satisfactory, in that a general property is inferred. The term "oil," as applied to these bodies, is a vestige of days when compounds were classified, owing to limited knowledge of their composition, according to their physical properties. Suffice it to say that these bodies are of complex composition and are waste products in the vital economy of the plants secreting them, but serve an important function as instruments of natural selection in repelling injurious insects and promoting cross-fertilization by pollen-carrying insects. The chief commercial value of essential oils resides in their perfume, which property is a direct result of their volatility, contact with the olfactory nerves being required for the production of odor. With volatile bodies difference of odor arises from difference of chemical composition. Owing to the complex composition of essential oils, even the superficial study of which would carry us into the intricacies of organic chemistry, an entirely satisfactory classification according to chemical composition is not possible. Essential oils are complex mixtures in which the odor-bearing body is not only present in varying proportions in the same oil, according to conditions of growth and manufacture, but is likewise present in oils of different botanical origin but of similar odor. As is frequently the case, odor is to be attributed not to a single body but to a mixture of them. Fractional distillation of essential oils indicates that they are composed of a nearly odorless vehicle consisting of one or more hydrocarbons of the terpene class, a principal odorous constituent which may be an alcohol, phenol, aldehyde, ketone, ether or ester, and small quantities of various other compounds. These modifying constituents vary in amount and character in oil obtained from different parts of the same plant.

Chemical Classification.—A botanical classification is more rational, but for the purpose in hand a chemical classification will more fully suffice to indicate the character of the respective odor-bearing bodies and their influence in the art of perfume blending. In a consideration of essential oils with respect to the sense of smell, distinction must be made between quality and penetrating power. Quality resides in the chemical composition of the body and those bodies of similar composition possess the same or a similar odor. It will be shown later, however, that in a few cases, differently consituted bodies possess a similar odor. The penetrating power of a body likewise resides in its chemical composition, but is greatly influenced by the condition in which it may exist. If by any means the volatility of a body is increased, its penetrating power is to that degree enhanced, as may be shown by its solution in alcohol and its reduction to a spray, whereby the evaporative surface is greatly increased.

Erdmann* classifies perfumes, or odor bearing bodies, according to their composition and the quality and penetrating power of their odors, into seven principal groups, in a descending order of the development of these characteristics, as follows: (1) Aldehydes; (2) alcohols and esters; (3) ketones; (4) phenols and phenolic ethers; (5) acids and acid anhydrides; (6) nitrogenous substances; (7) hydrocarbons.

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* Zeits. f. angew. Chem., 1900 [5], 103-116.

With regard to Group I, citral can be obtained by oxidizing the corresponding alcohol rhodinol (geraniol) or else the oil of linaloe. Furfural, one of the cyclic aldehydes, is always a constituent of clove-oil, in which it can be detected by the red coloration given with an acetic acid solution of β-napthylamine. Benzaldehyde is obtained artificially from toluene, while its next higher homologue, phenacetaldehyde (distilling at 90° C. under 15 mm.), which possesses a smell of hyacinths, can be obtained from cinnamic acid. It polymerizes very readily. Anisic aldehyde can be produced by methylating *p*-oxybenzaldehyde, but is usually obtained by oxidizing anethol. It possesses the odor of the flowering whitethorn (*Cratægus oxyacantha*), and comes into commerce in France as "hawthorn." The solid sodium bisulphite compound is also a commercial product. Cinnamic aldehyde is obtained technically from benzaldehyde. Vanillin, the essential principle of the vanilla pod, which contains about 2 per cent, is very widely distributed. After its constitution had been determined by Tiemann, it was originally obtained by oxidizing coniferin, but at the present time it is produced from eugenol obtained from clove-oil. This is first converted into isoeugenol, which is then acetylated and oxidized. No truly synthetic process for obtaining vanillin has been worked commercially, and in view of the low price of clove-oil and of vanillin itself, such processes have no prospects. Heliotropin, or piperonal, has a similar constitution to vanillin, and is obtained in like manner from piperin, the chief principle of pepper, but it can also be produced more economically from isosafrol. It is valued for its use in cherry blossom perfume.

Of the alcohols and esters (Group II), rhodinol, the constitution of which has been determined by Tiemann and Semmler,* is the principal ingredient of several important essential oils, as ottar of rose, geranium-oil, and citronella-oil. In a pure state it has a somewhat stale odor; the associated ingredients of the natural source being, therefore, essential to the sweet honey-like odor of

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* Berichte, 28, 2132.

TABLE XXIV—CLASSIFICATION OF PERFUMES ACCORDING TO COMPOSITION AND ODOROUS QUALITY.

1. ALDEHYDES.

Nos. 1 *and* 2, *open chain, unsaturated; No.* 3, *cyclic; Nos.* 4–11, *cyclic, benzene series.*

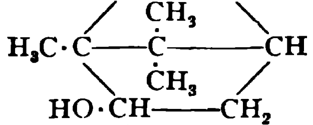
Name.	Constitutional Formula.	Occurs Native in
1. Citral	$(CH_3)_2:C:CH\cdot(CH_2)_2\cdot C(CH_3):CH\cdot CHO$	Oil of lemon, lemon-grass oil.
2. Citronellal	$(CH_3)_2:C:CH\cdot(CH_2)_2\cdot CH(CH_3)\cdot CH_2CHO$	Oil of citronella, lemon-oil, eucalyptus maculata.
3. Furfural	$\left.\begin{array}{l}CH{-}{-}CH\\ \dot{C}H\cdot O\cdot\ddot{C}\cdot CHO\end{array}\right\}$	Clove-oil.
4. Benzaldehyde	C_6H_5CHO	Oil of bitter almonds, laurel-oil.
5. Phenylacetaldehyde	$C_6H_5CH_2\cdot CHO$.	
6. Cinnamic aldehyde	$C_6H_5CH:CH\cdot CHO$	Oil of cassia, oil of cinnamon.
7. Cuminic aldehyde	$1\cdot 4\cdot C_6H_4\cdot C_3H_7\cdot CHO$	Oil of caraway.
8. Salicylic aldehyde	$1\cdot 2\cdot C_6H_4\cdot OH\cdot CHO$	Oil of spiræa, crepis fœtida.
9. Anisic aldehyde (hawthorn)	$1\cdot 2\cdot C_6H_4\cdot OCH_3\cdot CHO$.	
10. Vanillin	$4\cdot 3\cdot 1\cdot C_6H_3\cdot OH\cdot OCH_3\cdot CHO$	Vanilla, gum benzoin, balsam of Peru, beet-sugar.
11. Heliotropin	$4\cdot 3\cdot 1\cdot C_6H_3\cdot O\cdot CH_2O\cdot CHO$	Oil of spiræa.

2. ALCOHOLS AND ESTERS.

Nos. 1–4, *open chain, saturated; Nos.* 5–7, *open chain, unsaturated; No.* 8, *cyclic; Nos.* 9–13, *cyclic, terpene series; Nos.* 14 *and* 15, *cyclic, benzene series.*

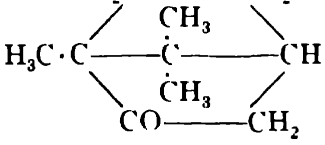
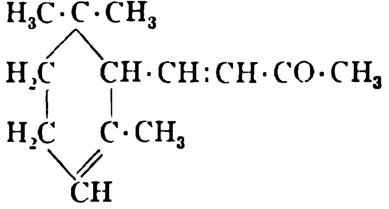
Name and Formula.	Combined with	Occurs Native in
1. Methyl alcohol, $CH_3\cdot OH$		Clove-oil.
	Benzoic acid (Niobe oil).	
	Salicylic acid (oil of wintergreen).	Gaultheria procumbens, Betula lenta.
2. Ethyl aclohol, C_2H_5OH	Formic acid (essence of rum).	
	Acetic acid (acetic ether)	White-wine vinegar, cognac, Magnolia fuscata(?).
	Butyric acid (pineapple-ether).	
	Isovaleric acid (apple-oil).	
	Pelargonic acid (artificial essence of cognac).	

2. ALCOHOLS AND ESTERS—(*Continued*).

Name and Formula.	Combined with	Occurs Native in
3. Isobutyl alcohol, $(CH_3)_2{:}CH\cdot CH_2\cdot OH$		Potato fusel-oil.
	Isobutyric acid	Camomile-oil.
	Angelic acid	Camomile-oil.
4. Isoamyl alcohol, $(CH_3)_2{:}CH\cdot CH_2\cdot CH_2\cdot OH$	Acetic acid (essence of pear).	
	Isovaleric acid (essence of apple).	
	Caprylic acid Caprinic acid } (œnanthic ether)	Oil of cognac.
5. Rhodinol (geraniol), $(CH_3)_2{:}C{:}CH\cdot (CH_2)_2\cdot C(CH_3){:}CH\cdot CH_2OH$		Otto of roses, geranium-oil, citronella, lemon-grass oil.
	Acetic acid	Oil of neroli.
6. Linalool, $(CH_3)_2{:}C{:}CH\cdot (CH_2)_2\cdot C(OH)(CH_3)\cdot CH{:}CH_2$		Linaloës, bergamot and lavender-oil, neroli, jasmine.
	Acetic acid	Bergamot-oil, neroli.
7. Citronellol $(CH_3)_2{:}C{:}CH\cdot (CH_2)_2\cdot CH(CH_3)\cdot CH_2\cdot CH_2OH$		Otto of roses, citronella-oil.
8. Furfuralcohol $\left\{\begin{matrix} CH\ \cdot\ CH \\ \ddot{C}H\cdot O\cdot \ddot{C}\cdot CH_2OH \end{matrix}\right\}$		Coffee-oil.
9. Borneol, $C_{10}H_{17}OH$		Borneo camphor, Ngai camphor, oil of rosemary.
	Acetic acid	Pine-tree oil.
	Isovaleric acid	Oil of valerian.
10. Terpineol, $C_{10}H_{17}OH$		Oil of cajeput, cardamom.
11. Menthol, $C_{10}H_{19}OH$		Oil of peppermint.
12. Eucalyptol (cineol), $C_{10}H_{18}O$		Wormseed-oil, eucalyptus-oil.
13. Peruviol, $C_{13}H_{22}O$		Balsam of Peru.
14. Benzyl alcohol, $C_6H_5CH_2OH$		Jasmine-oil.
	Acetic acid	Jasmine-oil.
	Benzoic acid	Balsam of Peru.
15. Cinnamic alcohol, $C_6H_5\cdot CH{:}CH\cdot CH_2OH$	Cinnamic acid	Storax.

3. KETONES.

Nos. 1 and 2, open chain, saturated; Nos. 3 and 4, open chain, unsaturated; Nos. 5–12, cyclic, terpene series.

Name.	Formula.	Occurs Native in
1. Methylamylketone	$CH_3 \cdot CO \cdot C_5H_{11}$	Clove-oil.
2. Methylnonylketone	$CH_3 \cdot CO \cdot C_9H_{19}$	Oil of rue.
3. Methylheptenone	$(CH_3)_2{:}C{:}CH \cdot (CH_2)_2 \cdot CO \cdot CH_3$.	Linaloes, lemon-grass oil.
4. Pseudo-ionone	$(CH_3)_2{:}C{:}CH \cdot (CH_2)_2 \cdot C(CH_3){:}CH \cdot CH{:}CH \cdot CO \cdot CH_3$.	
5. Carvone	$C_{10}H_{14}O$	Kümmel-oil.
6. Camphor	 (Bredt's formula)	Camphor-tree.
7. Fenchone	$C_{10}H_{16}O$.	Fennel-oil, thuja-oil.
8. Thujone (tanacetone)	$C_{10}H_{16}O$.	Thuja-oil, tansy-oil, oil of absinthe.
9. Pulegone	$C_{10}H_{16}O$.	Polei-oil.
10. Menthone	$C_{10}H_{18}O$.	Peppermint-oil.
11. Irone	$C_{13}H_{20}O$.	Orris-root (violet-root).
12. Ionone		

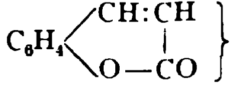
4. PHENOLS AND PHENOL ESTERS.

Nos. 1–9, *benzene series; No.* 10, *naphthalene series.*

Name.	Constitutional Formula.	Occurs Native in
1. *p*-Cresol methyl ether	$1\cdot4\cdot C_6H_4\cdot CH_3\cdot OCH_3$	Ylang-ylang-oil.
2. Guaiacol	$1\cdot3\cdot C_6H_4\cdot OH\cdot OCH_3$.	Beech- and birch-tar oil.
3. Creosol	$1\cdot3\cdot4\cdot C_6H_3\cdot CH_3\cdot OCH_3\cdot OH$	Birch-tar oil.
4. Anethol	$1\cdot4\cdot C_6H_4\cdot OCH_3\cdot CH{:}CH\cdot CH_3$	Oil of anise, oil of fennel.
5. Chavicol	$1\cdot4\cdot C_6H_4\cdot OH\cdot CH_2\cdot CH{:}CH_2$	Betel-oil.
6. Estragol	$1\cdot4\cdot C_6H_4\cdot OCH_3\cdot CH_2\cdot CH{:}CH_2$	Estragon-oil.
7. Eugenol	$4\cdot3\cdot1\cdot C_6H_3\cdot OH\cdot OCH_3\cdot CH_2\cdot CH{:}CH_2$	Clove-oil.
8. Thymol	$1\cdot3\cdot4\cdot C_6H_3\cdot CH_3\cdot OH\cdot C_3H_7$.	Oil of thyme (thymus vulgaris).
9. Safrol	$4\cdot3\cdot1\cdot C_6H_3\cdot O\cdot CH_2O\cdot CH_2\cdot CH{:}CH_2$	Sassafrass-oil, oil of camphor.
10. *β*-Naphthol methyl ether (nerolin)	$C_{10}H_7OCH_3$.	

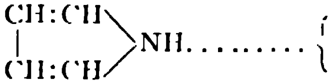
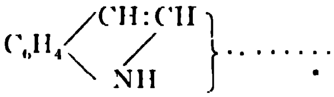
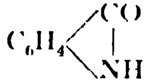
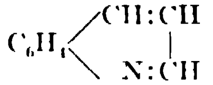
5. ACIDS AND ACID ANHYDRIDES.

Nos. 1 *and* 2, *open chain, unsaturated; Nos.* 3–5, *cyclic benzene series.*

Name.	Constitutional Formula.	Occurs Native in
1. Angelic acid	$CH_2{:}CH\cdot CH(CH_3)CO_2H$	Ester of camomile-oil.
2. Tiglic acid	$CH_3\cdot CH{:}C(CH_3)CO_2H$	Ester of camomile- and croton-oil.
3. Benzoic acid	$C_6H_5CO_2H$	Balsam of Peru, gum benzoin, balsam of tolu.
4. Cinnamic acid	$C_6H_5\cdot CH{:}CH\cdot CO_2H$	Balsam of Peru, of tolu, gum benzoin (Sumatra storax).
5. Coumarin	 $C_6H_4\left\{\begin{matrix}CH{:}CH\\ \vert\\ O-CO\end{matrix}\right\}$	Tonka beans, woodruff (asperula odorata).

6. NITROGENOUS PERFUMES.

Nos. 1–3, *open chain; No.* 4, *cyclic; Nos.* 5–12, *cyclic, benzene series.*

Name.	Formula.	Occurs Native in
1. Trimethylamine. .	$N(CH_3)_3$.	Chenopodium-oil.
2. Prussic acid . .	CNH.	Oil of bitter almonds, laurel-oil.
3. Allyl mustard-oil. .	C_3H_5NCS.	Mustard-oil, horseradish, Alliaria officinalis.
4. Pyrrol.		Derivative in orange-oil from the unripe fruit.
5. Indole.		Jasmine-oil.
6. Benzyl cyanide. . .	$C_6H_5CH_2CN$.	Oil of cress (Lepidium sativum, Tropæolum majus).
7. Mandelic nitrile.	$C_6H_5CH(OH)CN$.	Almond-oil.
8. Nitrobenzene (oil of mirbane). .	$C_6H_5\cdot NO_2$.	
9. Tonquinol (Baur's musk) .	$C_6H\cdot(NO_2)_3\cdot C_4H_9\cdot CH_3$	
10. Anthranilic methyl ester.	$1\cdot 2\cdot C_6H_4\cdot NH_2\cdot CO_2CH_3$.	Neroli, jasmine-oil.
11. Anthranil.		
12. Quinoline.		

7. HYDROCARBONS.

Nos. 1–10, *cyclic terpene series; Nos.* 11 *and* 12, *cyclic benzene series.*

d = *dextro; l* = *lævorotatory; i* = *optically inactive.*

Name.	Formula.	Occurs Native in
1. Pinene.	$C_{10}H_{16}$.	*d*-German turpentine, American turpentine, *l*-French turpentine.
2. Camphene.	$C_{10}H_{16}$.	*d*-Oil of ginger, oil of spike, *l*-citronella oil, valerian-oil.
3. Fenchene.	$C_{10}H_{16}$.	French turpentine (?).
4. Limonene.	$C_{10}H_{16}$.	*d*-Orange-peel oil, lemon-oil, bergamot-oil, *l*-oil of silver-leaved fir, *i*-(dipentene) oil of camphor.
5. Silvestrene.	$C_{10}H_{16}$.	Swedish, Russian turpentine.
6. Phellandrene. . . .	$C_{10}H_{16}$.	*d*-Water fennel-oil, elemi-oil, *l*-Australian eucalyptus-oil.
7. Terpinene.	$C_{10}H_{16}$.	Cardamom-oil.
8. Terpinolene . . .	$C_{10}H_{16}$.	
9. Cadinene.	$C_{15}H_{24}$.	Oil of cade.
10. Caryophyllene. . .	$C_{15}H_{24}$.	Clove-oil.
11. Cumene.	$1\cdot 4\cdot C_6H_4\cdot CH_3C_3H_7$. .	Oil of cumin, cyminum, thyme.
12. Styrene	$C_6H_5\cdot CH:CH_2$.	Storax.

attar of rose. Linalool is the chief ingredient of lily-of-the-valley perfume and occurs naturally in two different optically active forms together with linalyl acetate in bergamot, lavender, and orange-flower oils. Of the terpene series, oil of peppermint, of which menthol is the characteristic constituent, is one of the most important. Of the ketones (Group III), the cyclic compounds of the terpene series are the most important. This sub-group contains camphor, the crude product being obtained by distillation with steam, of the roots, branches, and twigs of the camphor-tree (*Cinnamonum camphora* or *Laurus camphora L.*); the liquid portion of the distillate constitutes oil of camphor, from which safrol is obtained by fractionation. Irone, the principle of orris-root (*Iris Florentine*), is isomeric with ionone. The latter is obtained commercially from citral by condensation with acetone, the first product being pseudo-ionone, which in turn yields ionone on treatment with dilute sulphuric acid.

The presence of guaiacol (Group IV) in birch-tar oil gives the latter the odor of Russian leather. Pure anethol forms 90 per cent of oil of Indian anise (badiane), and is the raw product from which anisic aldehyde is obtained. Thymol is employed principally for its disinfectant qualities, while eugenol, the essential principle of clove-oil, finds an extensive use for the manufacture of vanillin.

Natural Sources.—With the exceptions of musk and civet, which are secretions of the deer and cat of the same name, essential oils occur in varying quantities throughout the tissues of the plants secreting them. They are thus obtained from the root as oil of valerian; from the root and wood as oil of camphor; from the root and bark as oil of sassafras; from the wood as oils of cedar-wood and sandalwood; from the bark and leaves as oils of cinnamon and wintergreen; from the twigs as spicewood-oil; from the leaves as oils of eucalyptus and rosemary; from the undeveloped flower as oil of cloves; from the fresh flower as oil of lavender and rose; from the fruit as oils of caraway and anise; from the peel of the fruit as orange- and lime-oils; from the bark, twigs, leaves, and buds as oil of cassia; and from the entire plant as oils of thyme and peppermint. Others are obtained from grasses as oils of lemon-grass and citronella; and from balsams, resins, and gum resins as respectively storax-oil, frankincense, and elemi-oil.

Methods of Extraction.—Essential oils are obtained from their natural source by a variety of methods the use of any one of which for the extraction of any particular perfume will be determined by the stability of the body producing it and the state in which it occurs. The more delicate and elusive the perfume, the more refined must be the methods for its extraction; from which it will appear that the method employed for the extraction of oil of cedar-wood is not adapted for the separation of the perfume of jasmine. The various methods of extraction may be classified as follows: (1) Mechanical; (2) maceration; (3) enfleurage; (4) extraction with volatile solvents indirectly and directly; and (5) distillation.

Mechanical.—This is practicable mainly in treating the rinds of the citrus fruits (orange, lemon, etc.), which are considerable in bulk and contain oil in comparatively large quantities. One process consists simply in expression, the material being put into a press and subjected to heavy pressure. Another is to rub the fruit in a metal cup lined with spikes (écuelle à piquer), the oil settling into a hollow handle, whence it is at length poured. An écuelle on a larger scale, consisting of a drum lined with spikes, is also used. Another method consists in squeezing in the fingers sections of the peel turned inside out and taking up the oil with a sponge.

Maceration.—By this method the flowers are immersed in a bath of pure neutral fat as tallow, lard, or olive-oil maintained at the requisite temperature by means of a water-bath. Fresh flowers are added as the perfume of the previous charge is exhausted, and the latter removed, until the desired strength has been obtained. When a solid fat is used the saturated product is called a pomade; when a liquid fat as olive-oil, an *huile antique*. These perfumes when extracted from the fat vehicle with alcohol form the *extraits*, or floral pomade washings, of the perfume trade. When the flowers are allowed to remain too long in contact with the fat, the perfume suffers deterioration from the absorption of inferior odorous bodies from the flowers.

Enfleurage.—By this method extraction is effected at normal temperature, whereas by maceration a temperature of 65°–70° C. is employed. The apparatus consists of rectangular wooden frames about 24 inches long by 18 inches broad and 2 inches in depth.

In a groove all around the inside of each frame at half its depth a strong sheet of glass is inserted. The frames thus prepared are known as "chassis"; the separate "chassis" are laid upon each other to form a "pile."

On each surface of the sheet of glass in a "chassis" is spread a layer of prepared fat amounting to about 200 grams (7 oz.), which produces a coating some millimeters in thickness. These are "chassis à pomade."

There are also "chassis à huile," which consist of larger frames than the above, furnished on the inside with diaphragms of iron-wire netting instead of with sheets of glass. On the wire trellises are spread pieces of cotton cloth soaked in olive-oil of the very finest quality. These "chassis" are also placed one upon another to form "piles."

The fresh flowers are spread directly on the layers of fat, or on the oil-soaked cloths, and remain in contact with them for 24 hours; they are then removed and replaced by another batch of fresh flowers. This operation is repeated until the pomade or oil is saturated with the perfume to the desired extent, the process generally occupying two or three months.

When the desired degree of saturation is attained the pomade is removed from the "chassis" or the perfumed oil is expressed from the cloths by means of a hydraulic press.

Maceration and particularly enfleurage are employed for the most delicate odors as cassie, orange flower, heliotrope, jasmine, jonquille, muguet, reseda, rose, tuberose, and violet. These methods are slow and costly and give constant encouragement to the preparation of synthetic substitutes for their products.

Indirect Extraction with Volatile Solvents. — This method supplements extraction with non volatile solvents, viz., maceration and enfleurage. A pomade washer, Fig. 145, is employed for this purpose. This device is essentially a water-jacketed crutcher with detachable agitators. The lids are tight fitting and have a stuffing-box at the point where the agitator shaft passes through, so that there is no possible chance of evaporation of the material. The tanks are quickly detached from the shaft above when necessary to remove them from the frame. There is an opening in each lid for adding material while the machine is running. The gearing for each tank is provided with a clutch for starting and stopping the agitator. The operating mechanism is so arranged that the agitators are turned fifteen revolutions in one direction, then reversed automatically and run the same number of revolutions in the opposite direction.

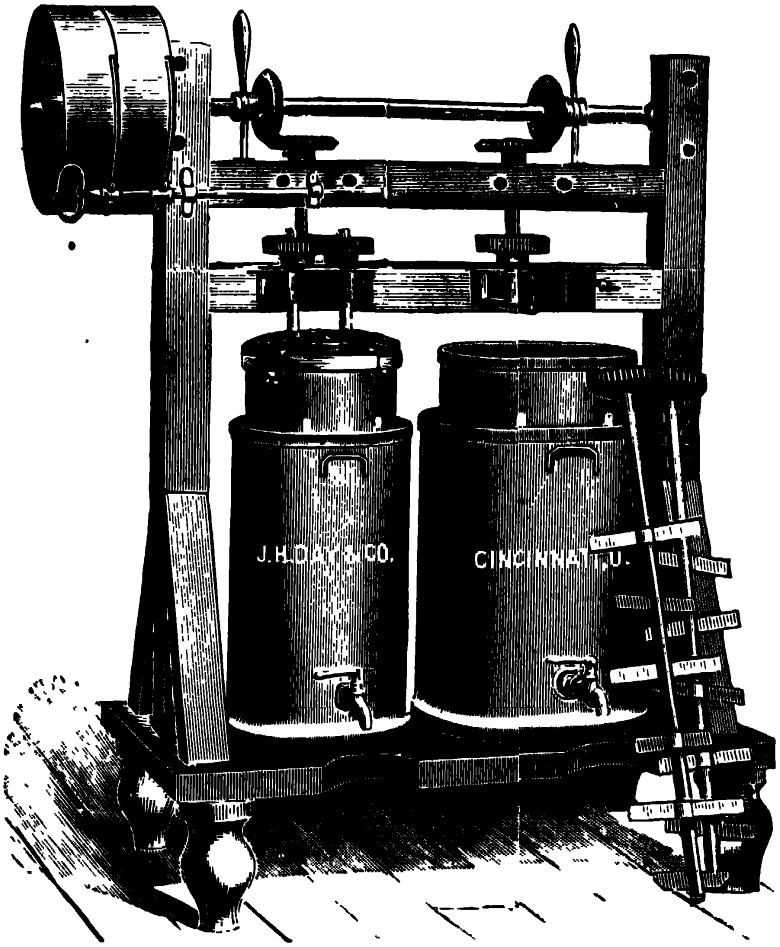

J.H.DAY & CO.
CINCINNATI, O.

Fig. 145.—Pomade-washer.

Extraction of perfumes from pomades by the use of alcohol is based upon the insolubility of the neutral glycerides, used as the absorbent of the perfume, in this solvent. Extraction therefore becomes a simple washing process. Second and subsequent washings, if made, are usually employed for the initial extraction of fresh batches of the same material. The washed pomade, which yet remains highly perfumed, may be used as a soap perfume, but is more commonly used as a cosmetic.

As a matter of fact,* a close examination of the old method of combining the floral odors with enormous masses of fat and offering them to the perfumer in the form of the so-called pomades gives an opening to the most severe criticism. For every manufacturer knows that the fat, when macerated or strewn over with flowers, will absorb the floral odor only imperfectly, and that washing with alcohol produces extracts of which the odor completely misses the fresh scent of the flower which they are supposed to reproduce. All pomade washes, without exception, suffer from an insipid, flat odor, caused by the particles of fat contained in them. The belief that it is possible to free the washes from these particles of fat by freezing is quite wrong, as notwithstanding the application of the most intense cold, particles of the fat (fatty acids) remain in solution in the alcohol, injuriously affecting the odor and the permanency of the extract.

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* Schimmel & Co., April, 1900.

Direct Extraction with Volatile Solvents.—All devices employed in this method work on the principle of a percolator, a subsequent step being the distillation of the solvent whereby the dissolved body may be recovered. It is evident that not only will the odor-bearing body be extracted, but also all substances soluble in the solvent used. Whether this process can replace extraction with non-volatile solvents or absorbents will be determined by the ease of purification of the concentrated product and the character of its odor. A modern extraction device for the operation of this process, wherein percolation and distillation are combined, is shown in Fig. 146.

This apparatus consists essentially of three receptacles, *A*, *B*, *C*, with which are respectively connected the cooling-worms, S^1, S^2, S^3, placed in the interior of a vat, *D*.

The three receptacles are connected through the intermedium of a pump and pipes properly arranged.

The receptacle *B* receives the liquid designed for drawing the perfume from the flowers, which are placed in a wire-gauze basket arranged in the receptacle *C*. A screw mounted below this basket, the centre of which is made hollow, causes a constant circulation of the dissolving liquid in which the flowers are immersed. The basket is refilled at intervals by raising the cover of the receptacle by means of the pulley suspended above it.

The receptacle *B* having been previously filled with the proper solvent (carbon disulphide, acetone, or petroleum ether), the valves of the three-way cocks R^1, R^2 are properly set to cause the liquid from the receptacle *B* to pass into the receptacle *C*, when the pump is started. The course followed by the liquid is indicated by the unbroken arrows. As soon as *C* is filled, the cocks, R^1, R^2, are closed, and the belt is shifted from the pump pulley to that on the shaft which controls the screw, thus setting the latter in motion. The exhaustion of the flowers then begins.

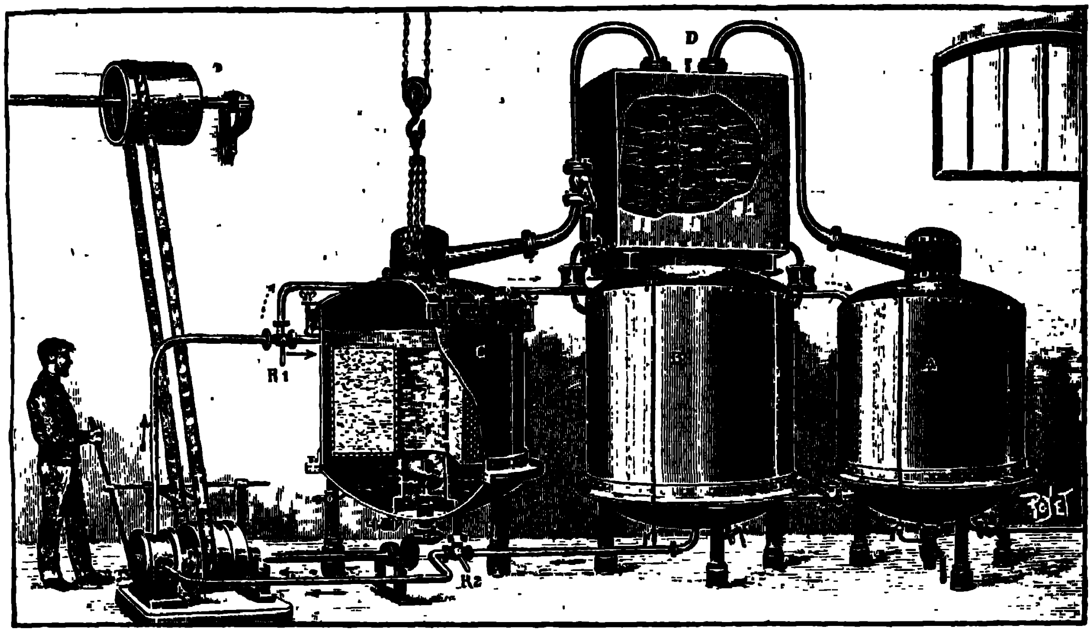

FIG. 146.—Arrangement of Extractors Employed by the Société des Parfums du Littoral at Frejus, France.

When this first operation is finished, the receptacles *C* and *A* are put in communication through the cocks R^1, R^2, and the liquid derived from the exhaustion is forced in the direction shown by the dotted arrows.

Having entered the receptacle *A*, the solvent is expelled by a current of steam and enters the reservoir *B* in a state of absolute purity, ready to be used for a second operation. The products of exhaustion remain in the receptacle *A*, whence they are easily removed. Upon the operation being several times repeated, the odoriferous substance contained in the flowers is completely extracted. At the end of the final operation, the material treated is heated by means of a worm in which steam circulates, so as to expel completely the liquid with which it is impregnated. Nothing is lost and the solvent is led back to *B* to be used again. In order to prevent any loss during the distillation, which may be watched through two small windows placed on the right and left of the vat, *D*, communication between the reservoir *B* and the atmosphere is established by a worm, S^2.

The crude product of the exhaustion is concentrated in a water-bath and purified carefully by appropriate methods.

Distillation.—The greater number of essential oils are extracted by steam distillation, direct fire being employed only with the most primitive apparatus. The process comprises (1) reduction of the raw material to a condition that will permit of the freest separation of the oil; (2) distillation in an apparatus especially constructed for the material to be treated and wherein steam may be used either saturated or superheated with closed coil, or with free steam and aqueous distillation, or these methods of application of steam singly or in combination in a continuous process or intermittently in partial vacuum; and (3) cooling and condensation of the vapor and purification of the crude distillate. The distilling apparatus consists essentially of a boiler or retort in which the raw material is placed, a source of heat, a condenser and suitable receiver for the collection and separation of the products of distillation.

When any plant or part of a plant is distilled by steam, a mixture of various chemical bodies will always distil over with the vapor, although the distilling material may not contain any essential oil whatever. Some of these bodies pre-existed in the plant itself as such, others have been formed by decomposition in the process of distillation. In cases where the distilling material contains an essential oil, the decomposition products of this oil are added to the chemical bodies referred to. Distillation products of this description are most undesirable bodies from the manufacturer's point of view, as they form impurities in the oil. Among them we * have found the following: Sulphuretted hydrogen, acetaldehyde, valeraldehyde, acrolein, ammonia, acetic acid, the lower and higher fatty acids, phenols, and hydrocarbons. By means of suitable processes of distillation the formation of these decomposition products may

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* Schimmel & Co., April, 1895.

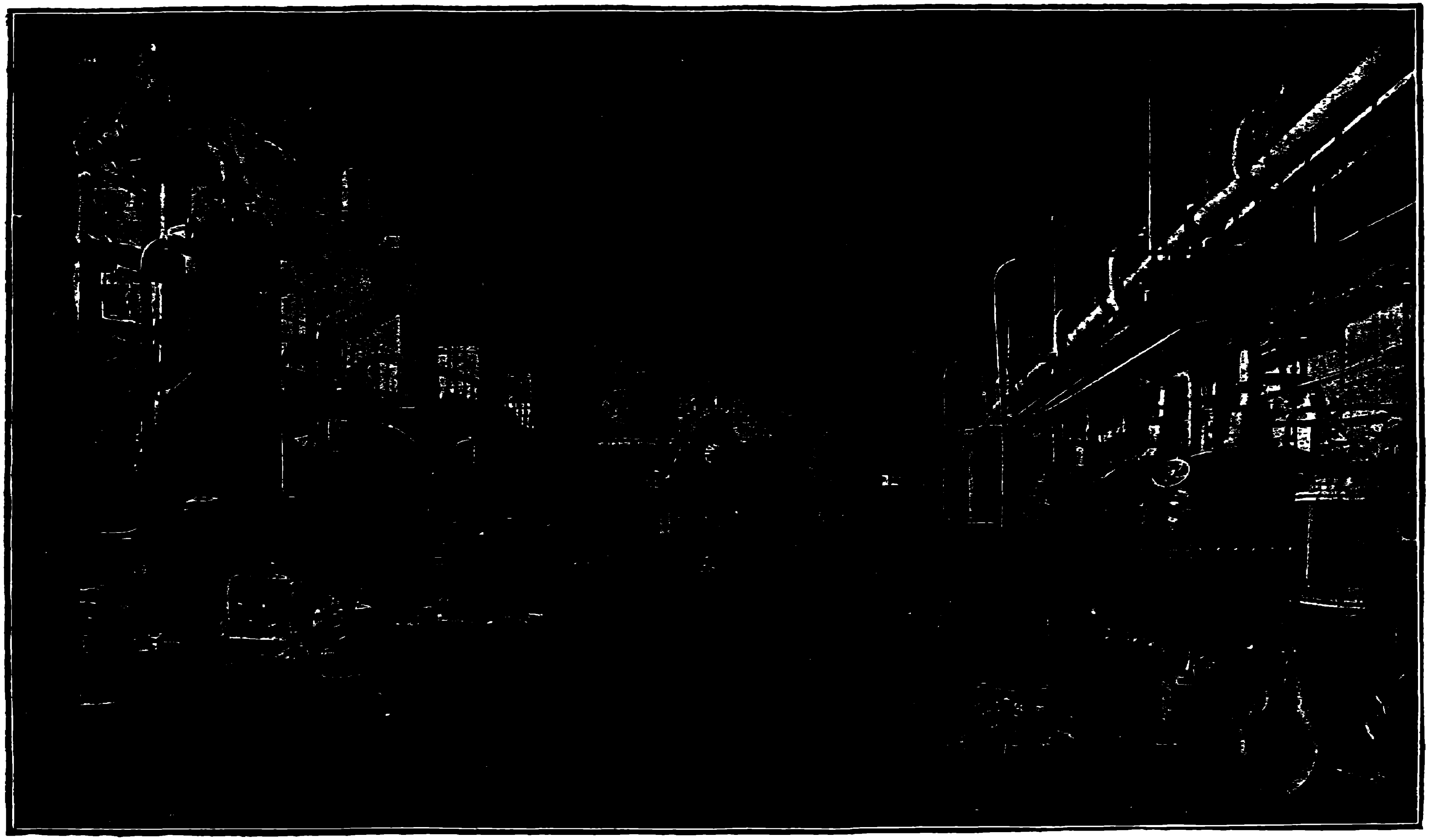

FIG. 147.—Distilling Apparatus for Essential Oils.

be greatly reduced; under favorable conditions they may be limited to an almost imperceptibly small residue, or they may be so altered, according to their characteristics, that they may easily be removed by subsequent rectification of the oil. We have also noticed, however, that these subsidiary products of distillation depend not only upon the distilling process and, naturally, upon the raw material, but that they vary to an exceptional degree according to whether the material is distilled in the fresh or the dried state. Distillation of the fresh material produces principally impurities in the shape of highly volatile, easily removable by-products (fresh leaves, for instance, mostly liberate currents of sulphuretted hydrogen), but in the distillation of dried material, even though it be distilled by steam in the same way as the fresh, empyreumatic bodies of a highly objectionable sharp and acrid odor, and a dark color (hydrocarbons, phenols, etc.) go over. This shows that there are certain vegetable materials which suffer chemical decomposition in the process of drying. The essential oils contained in them are likewise affected by these processes of decomposition, oxidation and polymerization, so-called "resinification" being the result.

Concentrated Essential Oils.—Fractional distillation of essential oils shows that they consist of a vehicle of inferior odor which usually contains two or more hydrocarbons of the terpene class (Group VII, page 385), and a principal odor-bearing body which may be represented by any of these present in Groups I-V, pages 381, 384, accompanied by smaller quantities of various bodies of indefinite composition. By subjecting essential oils, where practicable, to fractional distillation, the vehicle can be separated, leaving the remaining oil with an increased percentage of the characteristic odorous constituent. In this manner concentrated oils of anise, caraway, coriander, eucalyptus, lemon, etc., may be obtained. Concentrated oils are used chiefly in the preparation of liquers, syrups, and confectionery. They dissolve at once to a clear solution in spirit of lower strength than is required for the unconcentrated oil. When used in alcoholic solution in perfumery, the odor is not fully developed until after the alcohol has evaporated. It will be evident that in the preparation of oils of this type, synthetic bodies possessing odors of the characteristic constituents may be added to the original oil and in other ways great latitude allowed for the art of the compounder.

Substitutes for Essential Oils.—The difficulty of manufacture and high price of essential oils have encouraged their substitution by cheaper and, if possible, equally efficient products. This encouragement has been directly proportional to the choiceness of odor and price of the original body and the ease and cost of preparing the substitute. These substitutes form two general classes, viz., bodies isolated from essential oils and bodies obtained by synthesis either from coal-tar products or from bodies isolated from essential oils. In the former class may be included the bodies mentioned in the following list, which is not intended to be complete:

Isolated Body.	Chief Natural Source.
Safrol	Oil of camphor
Eugenol	Oil of cloves
Carvol	Oil of caraway
Cinnamic aldehyde	Oils of cinnamon, cassia
Citrol	Citrus oils, lemon, lime
Eucalyptol	Oil of eucalyptus
Geraniol	Oil of geranium
Linalool	Oil of linaloe
Menthol	Oil of peppermint
Anethol	Oil of anise
Turpentine	Crude turpentine (oleo-resin)

With the exception of the last, these bodies are representative of the concentrated oils mentioned in the preceding paragraph. Many isolated bodies, as eugenol, citrol, safrol, oil of turpentine, anethol, linalool, etc., serve as the basis of subsequent syntheses which, with the bodies mentioned, produce respectively—vanillin, the odor of vanilla; ionone, the odor of the violet; heliotropin, the odor of heliotrope; terpineol, the odor of the lilac; anisic aldehyde (aubépine), the odor of hawthorn; and linalyl acetate (bergamid), the odor of oil of bergamot.

Simple substitutes of coal-tar origin may be mentioned—as nitrobenzol (oil of mibane) and benzaldehyde, for oil of almonds;

methyl salicylate, for oil of wintergreen; cinnamic aldehyde, for oil of cassia; and methyl benzoate (oil of Niobe), with an agreeable balsamic odor. More involved syntheses from coal-tar products are required for the preparation of cumarin, the odoriferous principle of the tonka bean, which with suitable blending reproduces the odor of new-mown hay, and artificial musk, a cheap and more or less satisfactory substitute for the natural product.

All the artificial musks known in the trade are produced from homologous members of the benzene series which under the action of concentrated nitric acid give rise to dinitro or trinitro derivatives possessing a strong smell of musk.

Natural Musk, Civet, etc.—Artificial musk although used extensively is inferior to the natural product in delicacy and permanency of odor. Natural musk is the preputial secretion of the musk-deer (*Moschus moschiferous*), native to the Atlas and Himalaya Mountains. The composition of the body producing the odor is undetermined and is in no sense identical with that of the synthetic substitutes. The crude product occurs in the market in pods and grains in the following grades, named according to the habitat of the deer: Tonquin (Thibetan or Chinese), Nepaul, and Cabardine. Grain musk is that extracted from the pods and is more expensive than the pods.

Civet is a glandular secretion of the civet cat, native to northern Africa and the Indian Archipelago. It is of nauseating odor when in concentrated form, but when diluted, as in tincture, it is valuable as a binder or fixer of other perfumes.

Ambergris is a product of the spermaceti whale. It possesses a strong odor suggestive of musk and is handled as a tincture.

Gum benzoin is an exudation of the *Styrax benzoin*, a tree native to Sumatra, Java, and Siam. It has a very penetrating odor and is used to give body and permanence to perfume mixtures.

Adulteration.—The practice of adulteration of the volatile oils, which is probably as old as the manufacture itself, had, in the beginning, a certain justification, as with the incomplete technical equipment of the early times the addition of fatty oils, turpentine-oil, or alcohol was often necessary in order to extract from the plants their odorous principle. Later, when the preparation of the pure oils became known, the practice of making these additions was still retained.

Even thirty years* ago it was customary to distil coriander with the addition of orange-oil and to put the distillate on the market as coriander-oil. Since pure coriander-oil can now be prepared without difficulty, the product obtained by using orange-oil as it is found now and then, even at the present time, must be considered as adulterated, and if the foreign ingredient is not made known, its sale is a fraud.

The adulteration need not always be by the addition of a less valuable body; it sometimes consists in that the more valuable constituent of the oil has been partially removed. The effect is the same, whether from a caraway-oil of the specific gravity 0.910 so much carvol be removed that an oil of the specific gravity 0.890 remains behind, or whether the same result is attained by the addition of limonene to the same oil.

Although the adulterations themselves find a sufficient explanation, mostly in the profit and the pecuniary advantage to the adulterator, it cannot, however, be denied that often the ignorance of the consumer, and above all the desire to buy as cheaply as possible, is the cause of the spurious composition of many oils. More than once the producer may have been induced to adulterate, because he found no buyers for his pure products at a reasonable price, while his adulterating competitor was able to do a lucrative business at lower prices.

The main reason for the extensive adulteration to which volatile oils have been subjected at times is to be sought in the fact that the detection of adulterants was very difficult and often entirely impossible.

Owing to the growth of scientific knowledge of the terpenes and their derivatives great progress has been made during the last ten or fifteen years in the detection of adulterants. Knowing the composition of not a small number of volatile oils, it has become possible not only to distinguish between a pure and an adulterated oil, but also to judge the quality of these oils. This is effected by estimating the amount

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* "The Volatile Oils," compiled by Doctors Gildemeister and Hoffmann, under the auspices of Schimmel & Co., Leipzig, Germany.

TABLE XXV.—COMMON AND POSSIBLE ADULTERANTS OF ESSENTIAL OILS.

(Compiled by Dr. Geo. R. Pancoast and Lyman F. Kebler.)

Essential Oil.	Common and Possible Adulterant.
Almonds, bitter	Oils of apricot- or peach-kernels, alcohol, nitrobenzol, turpentine, and benzaldehyde.
Aniseed	Spermaceti up to 35 per cent, alcohol up to 80 per cent, kerosene, wax, oils of fennel, cedar, copaiba, camphor, turpentine, fennel, stearoptene, and oil of caraway (both from seed and chaff).
Angelica	Copaiba.
Amber, crude	Resin mixed with coal-oil and turpentine.
Amber, rectified	Resin-oil, turpentine, and kerosene.
Bay	Cloves, pimento, turpentine, and oils containing phenols. It has been adulterated with redistilled oil of cinnamon-leaf with slight admixture of redistilled oil of lemon-grass.
Birch	Methyl salicylate.
Bergamot	Lemon, orange, French turpentine, linaloe, and fatty oils.
Cajeput	Oils of camphor and turpentine. A mixture of rosemary- or savin-oils with camphor and resin of milfoil is often substituted.
Cajeput; Formosa	Said to be a mixture of cajeput and oil of camphor.
Camphor	Benzine, coal-oil, turpentine (one case 25 per cent).
Canada snakeroot	Copaiba.
Cananga	Cocoanut-oil.
Cassia	Coal-oil, fatty oils, resin (one case 18 per cent), oil gurjun balsam, cloves, cinnamon-leaf, cedar-wood.
Caraway-seed	Turpentine, oil of caraway-chaff, and added limonene. Term "twice rectified" for this article is rather misleading, as each rectification reduces per cent of carvol.
Cedrat	Mixture of orange and bergamot.
Cedar	Hemlock, spruce, turpentine, oil of camphor.
Cedar-leaf	Cedar-wood, thuja.
Celery-seed	Celery-leaf, turpentine.
Chamomile	Cedar, copaiba, turpentine, milfoil, lemon; lemon- or turpentine-oils sometimes distilled over chamomile-flowers.
Cinnamon	Cloves, cassia.
Citronella	Japanese oil of camphor (light variety), fatty oils, oil of gurjun, coal-oil, cocoanut-oil.
Coriander	Orange, cubeb, cedar, turpentine; oil of orange distilled with coriander.
Copaiba	Oil of gurjun balsam.
Clove	Clove-stems, fatty oils, copaiba, pimento, coal-oil, turpentine
Cubeb	Copaiba.
Curaçoa orange	Bitter orange and bergamot.
Dill	Oil of caraway-chaff, mace, turpentine.
Eucalyptus	Cheaper grades of eucalyptus. Turpentine is said to "smooth" a "rough" oil.
Fennel-seed	Fennel-chaff, alcohol, oils containing phenol.
Geranium	Ginger-grass, rectified citronella, fatty oils.
Geranium, Turkish	Fixed oils, turpentine, coal-oil.
Ginger-grass	Mineral oil, turpentine.
Hemlock	Spruce, turpentine.
Juniper-wood	Turpentine.
Lavender, garden	Spike, oil of camphor, turpentine.
Lavender flowers	Turpentine, alcohol, ester added to poor oil. Method of distillation frequently causes variations in specific gravity, optical rotation, and solubility.

COMMON AND POSSIBLE ADULTERANTS OF ESSENTIAL OILS—(*Continued*).

Essential Oil.	Common and Possible Adulterant.
Lemon.	Poor lemon-oil, with citral from lemon-grass added, poor old orange-oil, turpentine. When testing on paper, use fres lemon-peel for comparison.
Lemon-grass.	Fixed oils.
Limes, expressed.	Lemon.
Melissa.	Lemon, citronella, or lemon-grass distilled over melissa-leave mixtures of lemon and citronella and lemon-grass.
Matico.	Alcohol, turpentine.
Mace, distilled.	Poor quality nutmeg-oil.
Neroli.	Petitgrain with a little bergamot improves quality of a po oil. Lemon or orange increases optical rotation.
Orange.	Alcohol, turpentine. When testing on paper, use orange-pe for comparison.
Origanum.	A mixture of thyme, oil of camphor, turpentine, and colorin matter; crude oil of sassafras, rectified rosin-oil, Barbado tar, crude petroleum.
Palmarosa.	Cocoanut-oil.
Patchouli.	Cedar-wood, cubeb, turpentine.
Peppermint.	Mixture (peppermint, glycerin, alcohol, and turpentine copaiba, erigeron, turpentine, pennyroyal, alcohol, oil camphor, sassafras.
Pennyroyal.	Dementholized mint, turpentine, alcohol, residue from pe permint distillation.
Petitgrain.	Turpentine.
Pimento.	Cloves.
Pine-needle.	Turpentine. Much confusion exists in these oils, due part to nomenclature of Coniferæ.
Pinus sylvestris.	Scotch oil of fir, coal-oil, turpentine.
Rose.	Leaves of rose alba added to Bulgarian rose, as oil from th mixture contains more stearoptene, so that distiller is able add more geranium-oil without reducing melting-point b low minimum. Indian geranium or ginger-grass, palma rosa, true oil of rhodium, light paraffin oils, fixed oils, guaia wood oil, alcohol, spermaceti, paraffin.
Rhodium.	Mixture of rose and copaiba.
Rosemary.	Camphor and lavender, turpentine, spike-oil, alcohol, rectifi camphor-oil.
Rue.	Turpentine.
Sandal, "German". .	Mixture of sandal (English) and copaiba.
Sandal, "East Indian" or "English".	Copaiba, fatty oils, cedar-wood, oil of gurjun, West India sa dals.
Savin.	Juniper, turpentine.
Sassafras.	Safrol, oil of camphor.
Spearmint.	Turpentine.
Spruce.	Turpentine.
Tansy.	Spruce, turpentine.
Thyme.	Camphor, turpentine.
Verbena.	Lemon-grass.
Vetivert.	Fixed oils.
Wormwood.	Turpentine.
Wintergreen, true. . . .	Birch, pure methyl salicylate, and mixtures of the two.
Ylang-ylang (flower of flowers)	Cananga, fatty oils, synthetic oil.

of the principal or the most important constituent. In lavender-oil, bergamot-oil, petitgrain-oil, and others the amount of esters present is, therefore, determined; in thyme-oil, clove-oil, bay-oil, and Cretian origanum-oil, the amount of phenols is estimated; in cassia-oil and lemon-grass oil the amount of aldehyde. The assay of sandalwood-oil shows how much santalol, that of palmarosa-oil how much geraniol, is contained in the oil. The quality of the oils named finds numerical expression in the percentage strength of the active constituents, such as esters, phenols, aldehydes, and alcohols.

In a second class of oils, whose composition is likewise sufficiently known, an assay is not yet possible. The reason for this is twofold: first, the value of the oil depends not upon a single constituent, but upon the blending of the properties of several; and secondly, the chemical methods of investigation are not sufficiently developed.

With these oils the examination is restricted, as a rule, to the determination of the normal composition of the oil and the absence of commonly used adulterants. Such oils are lemon-oil, orange-oil, rosemary-oil, and spike-oil, which should be tested particularly for turpentine-oil.

Examination of Essential Oils.—Essential oils are tested for sophistication by physical and chemical methods, both varying according to the oil under examination. Physical methods comprise the following enumerated tests: Comparison of physical appearance and odor; determination of solubility, usually in alcohol of definite strength; determination of specific gravity; determination of congealing- and melting-points; optical rotation; refractive index; and separation of constituents of different boiling-points by fractional distillation. Chemical methods vary with the characteristic constituent or constituents of a given oil and may be classified as follows:

Determination of aldehyde by the bisulphite process. This method is applicable to aldehydic oils as those of Class I, page 381, of which oil of cassia is the chief representative. Determination of esters by saponification, applicable to the oils of Group II. Determination of alcohols by acetylization, also applicable to oils of Group II, and in the determination of total esters before and after saponification this method is combined with the preceding one. Determination of phenols by the caustic-potash method, applicable to the bodies of Group IV. Numerous other chemical tests are available for the detection of particular bodies as pinene, phillandrene, alcohol, fatty and mineral oils, for a more extended and detailed discussion of which, together with the natural history, chemistry, and manufacture of essential oils, the reader is referred to a recent exhaustive and very able work entitled "The Volatile Oils" (English translation by Kremers), by Gildemeister and Hoffman.

Blending of Perfumes.—The blending of perfumes is a recognized art which requires intimate familiarity not only with the chemical and physical properties of each essential oil, gum, resin, balsam, or animal ecretion, producing an agreeable and enticing odor, but with the modifying, or perhaps destructive, influence of each upon the other as they are compounded. The study of these properties by actual experiment and the harmonious blending of the various bodies in different proportions to produce different results, together with the preparation of the raw material, notably alcoholic extracts, in which condition all gums and animal odors, as well as the more expensive synthetic compounds and natural oils are used, requires a sum of knowledge and experience amply sufficient to give rise to the special art of the perfumer.

The perfume, after having been elaborated, must again be studied in connection with the article to be perfumed, or as is commonly the case, the article to be perfumed determines the character of the perfume used. The entire art is one of ceaseless experiment made more exacting by the popular demand for novelties. When prepared for direct sale as a perfume, the article consists of a volatile vehicle, commonly pure alcohol, diluted in varying proportions with water, in which is dissolved the odor bearing body. The latter is usually a mixture of bodies combined in such proportions as to most fully reproduce the scent or flavor of a particular flower or fruit, or to produce any distinctive odor necessarily agreeable. The odor-bearing body may be resolved roughly into three parts, viz., the distinctive odor, the modifiers, and the fixer. It is impossible to make any rational classification of odors. Rümner has attempted this in the following table in so far as he has grouped similar odors into a single class represented by a typical odor. The table is incomplete, but possesses some value in suggestion in the study of odors.

Class.	Type.	Odors of the Same Class.
Rose	The rose	Geranium, eglantine, palissander.
Jasmine	The jasmine	Lily of the valley, ylang-ylang.
Orange	Orange-flower, or neroli.	Acacia, syringa, orange-leaf.
Tuberose	Tuberose	Lily, jonquil, narcissus, hyacinth.
Violet	Violet	Cassis, iris, mignonette.
Balsamic	Vanilla	Balsams of Peru and Tolu, benzoin, storax, tonka bean, heliotrope.
Spicy	Cinnamon	Nutmeg, mace, allspice.
Caryophyllaceous	Clove	Pink.
Camphor	Camphor	Rosemary, patchouli.
Sandal	Sandalwood	Vetivert, cedar.
Citrine	Citron	Orange, bergamot, cedrat, lime fruit.
Lavender	Lavender	Aspic, thyme, wild thyme, marjoram.
Mint	Peppermint	Wild mint, basil, sage.
Anise	Anise	Aniseed, caraway, dill, fennel, coriander.
Almond:	Bitter almonds	Laurel, nut, mirbane.
Musk	Musk	Civet, musk-seed, musk-plant.
Amber	Ambergris.	
Fruit	Pear	Apple, pineapple, quince.

By a "modifier" is understood any additional odor or odors used to "soften," "temper," or "mellow" the distinctive perfume and is as variable in character and proportion as are the individual tastes of perfumers. Thus the oils of cedar-wood, cassia, lavender, rosemary, thyme, caraway, citronella, eucalyptus, etc., may be used to modify the odor of safrol, which constitutes the base of many perfume mixtures for laundry soap; whether the odor of the latter will be the distinctive odor depends upon the number, character, and proportions of the modifiers.

By a "fixer" or "binder" is understood any body or mixture of bodies, commonly used as alcoholic extracts, represented by ambergris (gray or black), balsams of Peru, Tolu, and storax, gum benzoin, orris-root (violet), vanilla (vanillin), glycerin, civet, and musk, employed to equalize the different rates of evaporation of different perfumes. Glycerin has been recommended to the extent of 4 per cent of the alcoholic solution to retard the evaporation of readily volatile bodies. When the fixer possesses a characteristic odor, as do all the others mentioned, it assumes the function of a modifier as well. Musk and civet in common with others mentioned possess in a high degree this property of harmonizing the different degrees of volatility of different perfumes whereby the liberation of the odors of the ingredients becomes more simultaneous and less of a successive separation. This property is manifested by such small proportions that the characteristic odor of the fixer may not be discernible, yet the body and permanence of the combined odor are increased.

Tinctures (infusions) of the fixers are obtained by trituration of the raw material with alcohol, the odors of which develop greatly on aging. With musk, exhaustion of the strength of the pod and the development of the odor are aided by the addition of a small proportion of carbonate of potash. The following is the strength of the more important tinctures as they are commonly purchased:

Tincture.	Proportion.	Relative Cost per Pound.
Ambergris (gray).........	4 ozs. to 1 gallon	$16.00
Civet..................	" " "	7.00
Gum benzoin (Siam).....	2 lbs. " "	1.50
Musk (Nepaul)..........	4 ozs. " "	14.00
Orris-root...............	6 lbs. " "	0.90
Vanilla (Mexican)........	1 lb. " "	4.00

Fixers may also constitute the base of a perfume mixture and those properties which militate against their separate use be counteracted by the addition of suitable modifiers. Fixers possess the property of persistence of odor arising from the exceptionally infinitesimal size of the particles separated in evaporation. When the characteristic perfume is sweet, an aromatic fixer may be used, as cloves, patchouli, vetivert, sandal wood, almond, vanilla, verbena, or ylang-ylang; where it is aromatic, tincture of storax, musk, civet, ambergris, or musk-seed may be used, the proportions varying with the preponderance of the typical scent desired.

Synthetic chemistry has supplied the perfumer with a large and increasing number of artificial odors which, while not always duplicating the natural source in intensity and delicacy, possess the advantage of greater uniformity of composition and lower cost. In addition to those already mentioned are benzyl cinnamate with the odor of balsam of Peru; bornyl acetate with the odor of fir-trees;

cinnamyl alcohol with the odor of hyacinths; salicylic aldehyde with the odor of meadow-sweet; geraniol (rhodinol) (limonol) with the odor of rose; geranyl acetate with the odor of lavender; and many other artificial compounds and secret mixtures with aromas duplicating many natural odors, particularly those of flowers, fruits, and plants.

Soap Perfumery.—Soap is perfumed either to mask an offensive odor associated with the use of inferior stock, as may be the case with low-grade laundry soap, or to harmonize agreeably with the natural odor, developed more strongly in use, of similar soap of superior quality. With soap made from better stock, as that used for soap intended for toilet purposes, perfume is added chiefly to increase the attractiveness of the product and to enhance the pleasure associated with its use. With so called medicated soap, possessing an odor characteristic of the medicament used, sufficient of the medicament is added to impart the odor, but impossibly in such quantities as to effect exclusively the curative results commonly claimed for such detergents. Soap of this character was discussed under Medicated Soaps.

The price of the soap in general determines the quality of the perfume used, but there are exceptions, notably in the toilet trade, where competition has to a degree been transferred from the detergent to the perfume and the package. And the rule that the best results in the use of perfume can be obtained only with the best stock has been modified by the ingenuity of the perfumer and the variety of brilliant dyes that have become available in recent years.

Perfuming Milled Soap.—The addition of perfume at normal temperature allows the use of a variety of material otherwise impracticable without loss by decomposition or evaporation, such as would occur with cold-process or boiled soaps, were it desired to add such material to them. The perfume mixture may be either compounded by the consumer from the stock of material kept on hand or it may be purchased from the dealer ready for use. Compounded perfumes, as a rule, bear names indicative of the odor, but are frequently used by the manufacturer without any reference to the name applied to the soap, or to the color, if any is used. If any consistency in this respect is observed, the perfume and color of the soap should suggest the natural odor and color of the flower whose perfume is imitated, likewise the name applied to the soap. The proportion of the mixture for milled soap varies from 8 to 32 ounces to 100 pounds of soap, according to the intensity of odor desired. It may be applied to the soap after having been reduced to laminæ by the preliminary milling, either in the box and well worked through by hand previous to adding the soap again to the mill or directly to the soap in the mill. With expensive perfumes it is advisable to reduce chances of loss by first incorporating it with some vehicle as pure vaseline or a portion of the laminated soap in a mortar and then adding the whole to the mass in the mill. The following perfume mixtures for milled soap are practical and reliable.

ORANGE BLOSSOM MIXTURE.

Neroli, artificial	3½ lbs.
Cumarin	1 lb.
Vanillin	⅛ "
Hyacinth	⅛ "
Oil linaloe, rectified	4 lbs.
" orange, sweet	1 lb.
Musk, artificial	¼ "
	10 lbs.

LILAC MIXTURE.

Heliotropin	4 ozs.
Oil cananga, Java	3 "
" ylang-ylang	1 oz.
Cumarin	5 ozs.
Oil geranium, reunion	5 "
" citronella, Java	1 oz.
" santal, East Indian	6 ozs.
" cedar-wood, Florida, extra	2⅞ "
Musk, artificial	⅛ oz.
Terpineol	180 ozs.
	208 ozs.

ALMOND-SOAP MIXTURE.

Oil of almonds, bitter, artificial, free from chlorine	64 lbs.
“ geranium, reunion	12 “
“ citronella, Java	4 “
Cumarin	4 “
Heliotropin	4 “
Vanillin	2 “
Solution musk, artificial, 1:100	10 “
	100 lbs.

GERMAN-COLOGNE MIXTURE.

Oil Neroli	15 lbs.
“ petitgrains, Paraguay	10 “
“ geranium, reunion	4 “
“ citronella, Java	1 lb.
“ sandalwood, East Indian	6 lbs.
“ cedar-wood, Florida, extra	4 “
“ lavender, I	5 “
“ bergamot	30 “
Solution cumarin, 1:10	20 “
“ musk, artificial, 1:100	5 “
	100 lbs.

WINDSOR-SOAP MIXTURE (LIGHT).

Oil lavender flowers, I	38 lbs.
“ caraway, Dutch	18 “
“ cassia	18 “
“ rosemary, Trieste	14 “
“ thyme, white, French	3 “
“ spearmint	3 “
“ cedar-wood, Florida, extra	6 “
	100 lbs.

EXTRA-FINE WHITE TRANSPARENT-SOAP MIXTURE.

Oil lavender, I	46	lbs.
“ geranium, renuion	17½	“
“ citronella, Java	5	“
Cumarin	10	“
Alcohol (cologne spirits)	21½	“
	100	lbs.

WINDSOR-SOAP MIXTURE (BROWN).

Oil lavender, I	32	lbs.
“ geranium, reunion	5	“
“ citronella, Java	2	“
“ cloves	20	“
Cumarin	1½	“
Oil cassia, filtered	24	“
“ caraway, German	8	“
Tincture gum styrax (2 lbs.: 1 gallon)	3½	“
Solution musk, artificial, 1:100	4	“
	100	lbs.

VIOLET SOAP MIXTURE.

Oil bergamot	16	ozs.
“ lavender, I	16	“
“ cedar-wood, Florida, extra	20	“
“ lilac flowers	4	“
“ geranium, reunion	4	“
“ orris root	1	oz.
Ylang-ylang	2	ozs.
Neroli	1	oz.
Ionone	1	“
Cumarin	2	ozs.
Vanillin	¾	oz.
Musk, artificial	¼	“
Oleoresin orris-root	8	ozs.
Gum styrax, liquid, strained	16	“
Balsam Peru	8	“
	100	ozs.

CHEAP VIOLET MIXTURE.

Safrol	6	ozs.
Oil bergamot	16	"
" lavender, I	24	"
" cassia	10	"
" geranium, reunion	12	"
" citronella, Java	2	"
" orris root	½	oz.
Balsam Peru	7½	ozs.
Gum styrax	8	"
Solution musk, artificial, 1:100	14	"
	100	ozs.

Perfuming Floating Soap.—A more delicate and lasting perfume is desired for floating soap than for laundry soap. Oil of lavender alone to the extent of 1.5–2.5 pounds per frame is very satisfactory. The following mixtures for white floating soap are employed in actual practice and produce good results.

No. 1.

Oil lavender	8	lbs.
" white thyme	2	"
" rosemary	4	"
" citronella	3	"

No. 2.

Oil rosemary, Trieste	1½	ozs.
" lavender, I	8	"
" thyme, white, French	½	oz.
Safrol	5	ozs.
Oil citronella, Java	1	oz.
	16	ozs.

No. 3.

Oil thyme	4.5	lbs.
" lavender flowers	9	"
" " aspic	9	"
" eucalyptus	3	"
" citronella	9	"
" rosemary	9	"
" caraway	4.5	"

The mixture in regulated amount is added to the soap in the crutcher just previous to discharging and is well worked through before framing.

Perfuming Laundry Soap.—With low-grade laundry soap, highly rosined or made from nigres, unbleached palm-oil to the extent of 15 pounds to the frame is a very efficient corrective of odor. It imparts permanency to the color of such soap and leaves no impression of cheapness produced by the use of a cheap and harsh essential oil or substitute. The following essential oils are commonly used as laundry-soap perfumes:

Oil lavender flowers
“ “ spike
“ cassia
“ cedar-wood
“ sassafras, artificial (safrol)
“ camphor

Oil citronella
“ eucalyptus
“ rosemary flowers
“ thyme
“ caraway
“ wintergreen
“ mirbane (nitrobenzol)

The proportion used is rarely in excess of 2.5 pounds per frame of soap, but may be increased as greater intensity is desired. A single essential oil may satisfy all requirements as oil of citronella or safrol. A good perfume for cheap laundry soap is found in a mixture of equal parts of carvene and citronella and cedar-wood oils. For a better grade the following formula produces good results:

Safrol	32 lbs.
Cedar wood	10 “
Cassia	5 “
Lavender flowers	2 “

The compounding of a perfume for soap of this character is a simple matter and consists in the preparation of a number of experimental mixtures using a fragrant and strong-bodied oil as safrol (art. oil sassafras) as a base, modifying it by the addition in varying proportions of any of the other oils mentioned. These experimental mixtures may be crutched in successive frames and the permanence and power of the perfume to harmonize with the natural odor of the soap observed on drying and in use. It is desired to produce the maximum harmony and permanence of odor at the least cost per unit of soap; this can be determined only experimentally by the method suggested.

Use of Colors.—Coloring-matter may be added to soap to conceal the inferiority of the stock, as in the use of unbleached palm-oil in cheap laundry soap, or to produce the impression that the soap has been made entirely or in part of the stock whose color persists in the finished soap, as in many so-called palm-oil and olive-oil foots soaps in which the oil has been used fort his purpose; to retard deterioration in appearance, as in the use of chlorophyll compounds to counteract the appearance of yellow blotches in soap containing cottonseed-oil; to heighten the natural color, as in the use of small quantities of ultramarine blue in white floating soap; to impart certain physical properties, as in the addition of ultramarine blue (bluing), to laundry soap (this may also serve to conceal an inferiority of stock); to impart so claimed hygienic properties, as in the use of pine tar, carbolic acid, etc.; as a simple dye, as in the soap compound for dyeing fabrics, or in harness soap; to impart a certain physical appearance, as in the use of copperas ($FeSO_4$) or ultramarine in the manufacture of mottled soap; or primarily to impart beauty, as in milled soap, or to conceal inferiority of raw material, as in cheaper detergents of the same class.

Organic Colors.—As simple dyes, mineral colors as well as those of vegetable origin, have been practically displaced by the more brilliant and cheaper water- and alcohol-soluble and alkali-proof dyes furnished by the substitution products and homologues of benzene, naphthalene, and anthracene, obtained by the fractional distillation of coal-tar. Coal-tar dyes are classified into a number of groups, according to their origin and properties. Each group produces a variety of colors known to the consumer by fanciful or descriptive trade names. Every desired shade of red, pink, orange or yellow, green, brown, blue, or lilac may be procured from dealers, it being necessary only to decide upon the color, and the same can be procured with full and most improved directions for use.

Safron, annatto, turmeric, cochineal insect (carmine), etc., have been largely dislpaced by the coal-tar dyes. Chlorophyll compound has come into use for imparting a green color to soap and to some extent to edible oils in imitation of olive-oil. Chlorophyll itself is a body of very complex and unstable constitution, but under the action of acids and alkalis it yields bodies of remarkable stability. Phyllocyanin, obtained from chlorophyll by treatment with strong acids, yields with metallic oxides bright-green compounds of great stability. Chlorophyll compound occurs in trade as a dark-green viscous or syrupy mass.

Inorganic Colors.—The prime requisites of a soap color are solubility in water, inertness towards alkali and permanency under the conditions obtaining in the use of the product. Inorganic or mineral coloring-matters, therefore, find very limited application in the soap industry, their sole use being as a mottling agent. The chief colors used for this purpose are copperas (Fe_2SO_4), iron oxides known variously as brown and red ochre, venetian red, burnt sienna, indian red, etc., and the different ultramarine colors.

Ultramarine is a double silicate of sodium and aluminum, together with sodium sulphide, characterized in a fine state of division by brilliancy of color and high covering power. Concentrated or even dilute mineral acids decompose all kinds of ultramarine with evolution of part of the sulphur as sulphuretted hydrogen and precipitation of the remainder. Stronger organic acids have the same effect. Alkaline solutions, hot or cold, do not alter green or blue ultramarine, but violet becomes blue. There is sulphate ultramarine and soda ultramarine, according to whether in its production sodium sulphate or soda is used. The raw materials from which ultramarine is prepared are either clay (kaolin), sodium sulphate and carbon, or clay, soda, sulphur, and carbon; in the latter process a small quantity of pure sand is added to the ingredients. The very finely ground and intimately mixed substances are subjected in fire-proof clay pots to a strong red heat, whereby, in case sodium sulphate is used, the so-called ultramarine green is produced, which is much prized and employed by painters as a pigment, proof to lime and light. This mass is now roasted at a low temperature, with addition of a little sulphur, under the action of air, and thus the blue ultramarine is obtained. The process of this transformation is very simple, but it is rendered superfluous and a blue product is obtained outright by using soda instead of sodium sulphate. The soda ultramarine is darker than the sulphate ultramarine. A number of shades occur in commerce, which with pale varieties are obtained by the addition of kaolin.

Application of Colors.—The color may be applied, preferably dissolved in water, for crutched soap or in alcoholic solution mixed with the perfume for milled soap. In the latter case, if applied in the dry state, it may be simply dusted upon the soap as it is being worked in the mill. As many colors admit of solution in essential oils, it may be advisable to prepare a considerable quantity of the perfume in advance in this manner. Preliminary solution is made with a small quantity of the oil, which is afterwards mixed in regulated amount with a larger volume. The intensity of the color depends upon the amount used, 1 ounce per 100 pounds of soap being generally sufficient. Aniline colors attain their highest brilliancy with soap of the smoothest texture, particularly where cocoanut-oil is a stock ingredient. With aniline colors particularly sensitive to alkali the best results can be secured only with neutral soap; this limitation, however, has been largely removed, the only disadvantage being deterioration on long exposure to sunlight.

In the following table is given a list of colors and the commonly used material employed for producing them in soap.

Color.	Material.
Red	Coal-tar dyes, as fuchsine, rhodamine, and eosine; combinations of violet and orange.
Orange and yellow	Coal-tar dyes; combination of red and yellow for orange, and orange and green for yellow.
Green	Coal-tar dyes; ultramarine green; chlorophyll compound; combinations of blue and yellow.
Brown	Coal-tar dyes; combinations of blue and yellow; caramel.
Blue and lilac	Coal-tar dyes; ultramarine blue; combination of violet and green for blue.
Violet	Coal-tar dyes; combinations of red and blue.

In all cases in using colors they should be completely dissolved, otherwise the soap will become spotted. To avoid this it is advisable to strain the solution, usually 1 part of color to 50 parts of water, through cloth and the clear filtrate used.

With settled soap, to which the coloring-matter is added invariably in the crutcher, whether the soap on the addition of the solution of coloring-matter is hotter or less hot in one case than in another is of no consequence. It is, however, self-evident that in order to obtain a uniform coloration of the soap the coloring-matter must be evenly and thoroughly distributed, and no pains should be spared to accomplish this.

With soaps made by the cold process the solution of the coloring-matter is first added to the oil and thoroughly mixed with it. To the oil thus colored the alkali is then slowly added with active stirring and the stirring continued until the soap which drops from the stirring-apparatus accumulates on the surface. It is only when this takes place that the perfuming of the soap should be effected.

With milled soaps, which are mostly produced in delicate shades of color, the most scrupulous cleanliness should be observed. Before the beginning of the milling process the machinery should be most carefully cleaned. In the actual milling process the soap is allowed to pass once in a dry state through the rollers. It is afterward somewhat moistened, the respective solution of coloring-matter poured upon it, and thoroughly worked with the hands. The colored soap is now repeatedly passed through the rollers until the coloring-matter is thoroughly mixed with the soap, and so completely distributed that the original color of the plain soap can no longer be seen. It is only when this has been accomplished that the soap should be perfumed.

With transparent soaps the solution of coloring-matter is added when the soap is finished and has cooled to about 55° to 60° C. (131° to 140° Fahr.). A good distribution of the color by a sufficient manipulation of the soap is an essential condition for obtaining a handsome product.

In the preparation of transparent soaps which contain sugar care must be taken to avoid all unnecessary heat, as otherwise, on account of the burning of the sugar, the soap itself will easily acquire a dark color, and the coloring-matter, which is subsequently added, will then not have its full effect. The perfume is added directly after the coloring has been effected.

Filling material, especially silicate of soda, affects colors in a most unfavorable manner, producing not only discoloration, but effects entirely different from those desired. It is rarely possible to impart to a filled soap a handsome, fiery shade of color.

CHAPTER XII.

MILLED SOAP.

Milled Soap. Historical. Preparation of Soap for Milling. Mixing Ingredients. Mills. Operation of Mill. Plodders. Operation of Plodder. Pressing. Continuous Method.

Milled Soap.—As previously stated, milled soap is grained, settled soap from good stock which has undergone a partial dehydration, thorough kneading, and compression into an elongated bar which is cut into cakes of suitable size and pressed. Soap made in this manner possesses distinctive qualities of durability, mildness, and beauty which make it for toilet purposes superior to ordinary settled soap. The character of the ingredients and method of manufacture of the soap-base for milled soap have already been described. To be a milled soap of best quality it must be made from the best materials saponified and perfectly neutral, without addition of inert matter of any description, homogeneous, lathering freely in hot or cold water, but must not waste rapidly in hot water, must not discolor to any extent on aging, nor contain matter other than perfume that will injuriously affect the skin. The requirements of the U. S. Navy Department for a milled toilet-soap are as follows:

To be milled, neutral, soda soap, made from clean, wholesome fat and as free as possible from water, rosin, and mineral, starchy, or foreign material. Analysis must show not more than three-tenths of 1 per cent of mineral matter, three-tenths of 1 per cent of carbonated alkali, calculated as carbonate of soda (Na_2CO_3), one-half of 1 per cent uncombined alkali, calculated as caustic soda ($NaOH$), 1 per cent common salt, or 14 per cent of water. A cylinder of soap seven-eighths of an inch in diameter and 1 inch high cut from a cake must sustain a weight of 15 pounds for five minutes without crushing or compressing more than one-sixteenth of an inch. Soap will be rejected if made so largely of cocoanut-oil, palm-oil, or other fat of characteristic smell that the peculiar odor remains on the hands after using. To be perfumed with the characteristic odor of lavender; perfume to add not more than 5 cents per pound to the cost of the soap.

Cakes to be oval, to weigh about 4 ounces; color, light brown. Each cake to be wrapped in soft paper; to be packed in neat paper boxes, three cakes to a box.

The soap will be bought by the pound.

For inspection, one cake taken at random will be examined, and the lot will be accepted or rejected on this sample cake.

The weight of the soap to be paid for will be determined by the amount of combined alkali or its equivalent in the lot; this to be found by multiplying the weight in grains of combined alkali in the sample cake by the number of cakes, then dividing this product by 630, the number of grains of combined alkali assumed as a standard pound of soap.

Historical.—The original manufacturers of milled soap were the French, whose product early attained a popularity which persists to-day, notwithstanding the fact that soap of equal quality is produced elsewhere. The prestige of the French in this respect was greatly aided by their skill as perfumers; but equal proficiency in this direction, as well as in the manufacture of milled-soap machinery, have displaced them from the preëminence which they so long occupied. The early manufacture of milled soap was naturally very primitive, the dried shavings being reduced and mixed with perfume by means of a mortar and pestle and the agglomerated mass moulded into bars by the pressure of the hands. Subsequent mechanical devices have not departed from this simple procedure.

Milled soap was first made in the United States in Philadelphia in 1844 by Jules Haul, a Frenchman, who in that day kept a shop for the sale of imported soaps and perfumes. He began making toilet-soaps in a small way in the rear part of his store with a double iron roll sugar-mill and an old screw-press, both worked by hand. The dried chipped soap was passed between the rolls a number of times until homogeneous. It was then shaped by hand with a wooden paddle to as near the dimensions of the die as possible, after which the bars were laid away on racks for two or three weeks until well dried. They were then pressed by means of the screw-press into a plain die without lettering, after which they were again dried for two or three weeks, and were finally pressed in a die with lettering. It required a period of four or five weeks before the soap was ready for sale.

This method of manufacture continued until 1847, at which time A. W. Harrison, of Philadelphia, imported from France a set of toilet-soap machinery which consisted of a two-roll granite mill and a plodder. The plodder consisted of a horizontal cylinder about 8 inches diameter and 24 inches long. The soap was placed in the cylinder and compressed with a screw, after which the soap was allowed to remain, with the pressure applied, for several hours. This machine, being of light construction, did not admit of the application of heavy pressure, with the result that the soap was not very compact. This plodder produced about 100 to 150 pounds per day, which was not only quite an increase over the quantity made by hand, but the soap had a much finer appearance.

Jules Haul, having become aware of this formidable source of competition, contracted with Sigmund Rutschman, a machinist, who had installed and repaired the Harrison plant, for a duplicate set, which was duly set up with improvements upon the French pattern in the autumn of 1847. Messrs. R. & G. A. Wright, of Philadelphia, had a duplicate set of toilet machinery made and began the manufacture of milled toilet-soaps in the following year.

In 1854 M. X. Bozine, of Philadelphia, increased the equipment of milled soap machinery, previously supplied by Rutschman, by a three roll mill and a vertical plodder imported from France. The plodder was of heavier construction and greater capacity than any hitherto used, permitting greater compression, resulting in a more compact and handsomer product. In 1870–1873 milled-soap plants were installed in New York City. In 1877 1878 a hydraulic plodder of a capacity of 200–250 pounds of soap per charge and with pressure of 4000 5000 per square inch was introduced into a number of large manufactories.

A type of vertical hydraulic plodder, now of only historical interest, is shown in Fig. 148. The hydraulic plodder, while providing all that could be desired in compressive force, was intermittent in action, made necessary by recharging, whereby the output of soap with such a machine could not be adapted to increasing demands made upon it. A continuous plodder was devised which consisted of an Archimedian screw enclosed by a cylindrical case, the principle of its construction being shown in Fig. 149.

The operation of this type of plodder, being continuous, provided a much larger output than was possible by the use of the intermittent hydraulic plodder, but required, however, that the soap be in a softer state, as compression depended entirely upon friction of the soap against the cylinder in which the screw was encased. It was found that increased compression could be obtained by the use of a device consisting essentially of two spiral convergers arranged parallel within a casing on opposite sides thereof constructed of two bodies securely bolted together. By this arrangement of twin screws, it was claimed, a degree of compression almost twice that of a single-screw plodder could be obtained. The principle of construction of a double-screw plodder is shown in plan, elevation, and section in Fig. 150.

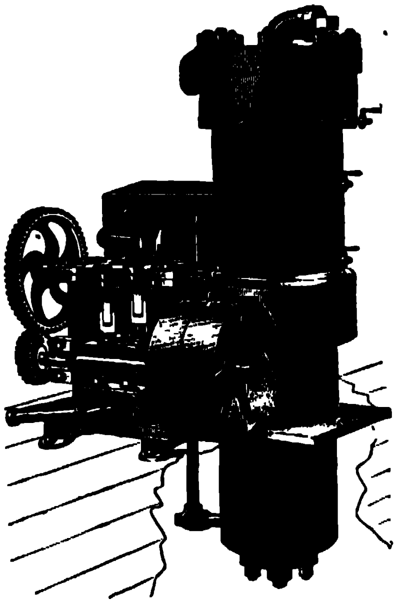


FIG. 148.—Vertical Hydraulic Plodder (1882).

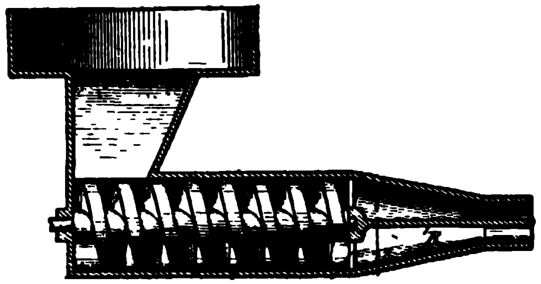


FIG. 149.—Section of a Single-screw Plodder, showing Principle of Construction.

Preparation of Soap for Milling.—The boiling of the soap-base for milling has been already described and requires no further comment at this place. After cooling in the frames, the soap is stripped, slabbed, and cut into long narrow bars. For this purpose the first cutting-head of the cutting-table is removed whereby the width of the frame is made equivalent to the length of the bar. Previous to slabbing, the sides of the frame are scraped to remove any discoloration arising from contact of the soap with the iron frame-sides. The bars are racked and allowed to dry. Soap made from stock suitable for milled soap dries rapidly and is soon ready to be reduced to chips. For this purpose the ordinary chipper, as described in the manufacture of chipped soap for laundry use, is employed. Freshly cut soap contains upwards of 30 per cent of water, which is reduced from 3 to 5 per cent in passing through the drying-room. In the drying of milled-soap base the amount of water is reduced almost one-half, viz., to 15 to 18 per cent. This proportion varies according to the stock used and the quality of soap desired, being determined to a large degree by the way in which the soap behaves under the subsequent mechanical treatment in the mill and plodder. Initial drying is to prepare the soap for chipping, after which with the large extent of drying surface proper conditions for milling are readily obtained.

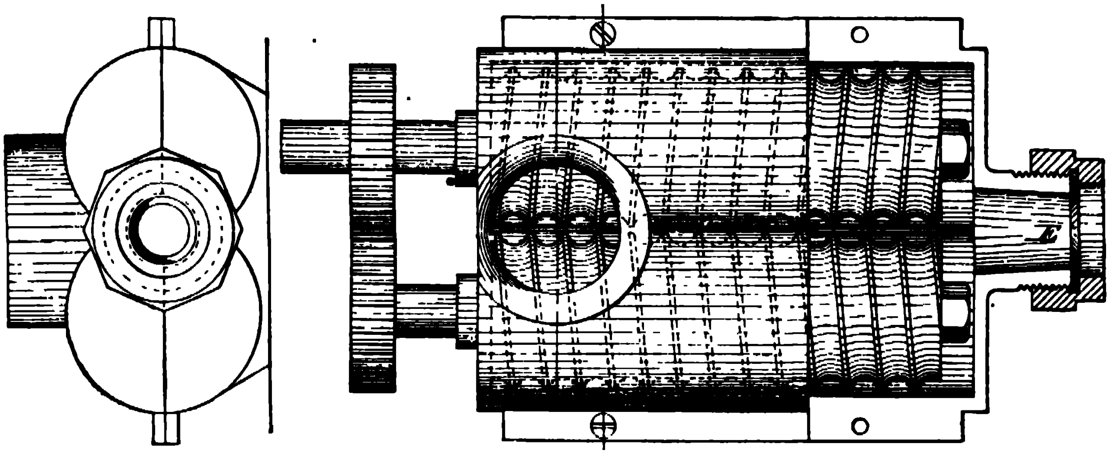


FIG. 150.—Double-screw Plodder, showing Principle of Construction.

The method of handling chipped soap during drying varies, a common procedure being to discharge the soap from the chipper directly into shallow trays with bottoms of galvanized-iron wire netting. These as filled may be stacked on trucks and conveyed to the drying-room, or stacked in piles and allowed to dry spontaneously. The proper degree of dryness is determined by experience and, with soap made from any given stock or intended for finished soap of a given quality, depends upon whether the soap is finished pure or with the addition of inert matter, usually talc, which possesses moisture-absorbent properties. With soap made from cocoanut-oil and tallow and milled pure, less drying is required than with soap made from softer stock such as may contain more or less cottonseed oil or rosin; likewise less drying is required when soap made from the former stock admixture is to be milled with talc. Excessive drying may be readily corrected by the addition of water during the milling process, or preferably before milling in a machine made for the purpose of amalgamating perfume with the dried chips. Excessively dried soap is of short texture, more or less brittle during milling, and during plodding, if not softened thereby, tends to crack as it is discharged. Insufficiently dried soap to be finished pure under no circumstances should be milled, as satisfactory results at no stage can be obtained. It adheres unduly to the rolls of the mill, requiring prolonged milling to insure perfect homogeneity and then will be more or less streaky and smeary. By the addition of talc, or starch, which is common with cheaper grades of milled soap, or by the addition of drier soap, the excess of moisture is taken up and better results secured.

Mixing Ingredients.—With the soap in proper condition to mill it may be mixed with perfume and coloring-matter, or talc if the latter is used, in a zinc-lined box by hand, and the whole then transferred to the hopper of the mill. An improved device for mixing ingredients with the dried soap consists of a mechanical amalgamator, shown in Fig. 151, which breaks up and amalgamates the chips, perfume, and coloring-matter prior to milling. By thorough mixing of the ingredients before milling, the milling period is shortened, one or two millings sufficing to secure perfect homogeneity. The mechanical amalgamator eliminates the hand labor required for mixing and secures the most thorough mixing in a short time. It is built to reverse automatically as shown or to run in one direction only. The body of the machine tips over to permit emptying into the regular milling-boxes.

Mills.—The toilet-soap mill consists essentially of a substantial

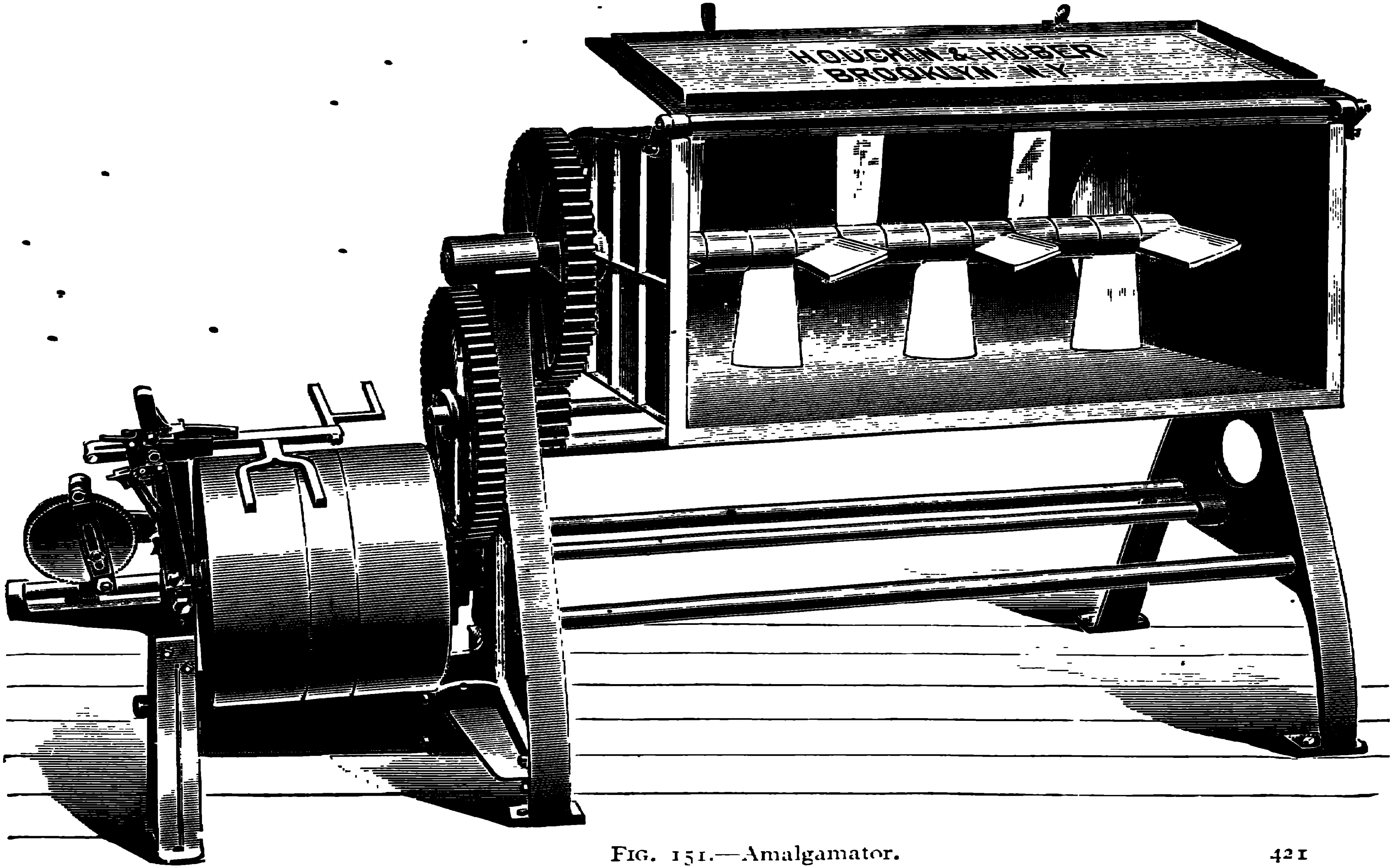
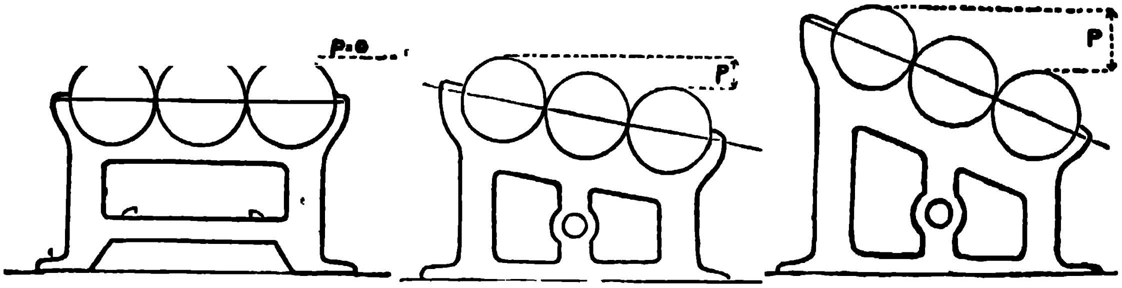


Fig. 151.—Amalgamator.

iron frame with bearings in which are mounted shafts carrying polished and non-absorbent cylindrical granite-crushing rolls arranged horizontally or with a pitch, or in a combination of these positions. Variations of pitch are shown in Fig. 152, by pitch being understood the difference in height between the tops of the extreme rolls. The rolls are made of close-grained Quincy granite securely mounted upon heavy steel shafts and adjusted by a worm and gear, whereby a parallel motion and even-wearing surface of the rolls are provided. As a rule the rolls vary in number from 3 to 5, although in the great size mill shown in Fig. 154, eight rolls are used. They vary in diameter from 12 to 18 inches and in length from 2 to 3 feet. The method of applying power and the construction of the driving-gear, whereby adjacent rolls have different rates of speed, will be evident from a study of the illustrations. A hopper is mounted over the feed-rolls, to which power is applied first and indirectly by means of suitable gears to adjacent rolls. A two-stone mill is shown in Fig. 153.



Mill with rollers in one plane. Low-pitch Mill. High-pitch Mill.

FIG. 152.—Diagram showing Variation in the Pitch of Toilet-soap Mills.

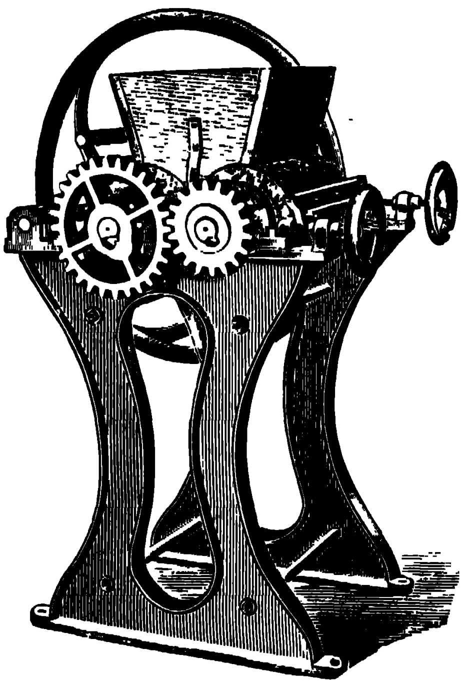


FIG. 153.—A Two-stone Mill.

Operation of Mill.—The purpose of milling is to reduce the dried chips with perfume and coloring-matter, or talc if used, to a perfectly homogeneous mass. The chips may be passed through the mill once and then mixed with perfume and coloring-matter, or they may be mixed before milling by use of the amalgamator, the same machine being used whatever procedure is employed. Reduction of the soap alone to a fine state of division by a preliminary milling admits of a more thorough incorporation of the perfume and color on subsequent passages through the mill. The soap is removed from the last roll in thin ribbons by means of serrated knife-edges adjusted against the roll. During the preliminary milling the rolls are adjusted to the coarseness of the material, and as this is reduced on subsequent treatments, closer adjustment of the rolls is made. The number of millings usually given the soap depends upon its quality, ease of incorporating the ingredients, and number of the rolls; for cheaper grades 3 to 4 millings suffice, while for the finest grade 7 to 8 millings are common. The capacity of the mill is, therefore, variable. A three-roll mill with rolls 18″×24″ has a capacity of about 200 pounds per hour, while the eight-roll mill, Fig. 154, has a claimed capacity of 3.5 tons daily and requires but one passage of the material. It consists essentially of a series of rolls arranged on an inclined plane, and formed of successive couples or pairs of rolls arranged one in front of the other, and with scrapers for the rolls, said scrapers being adapted and arranged to automatically transfer from one couple of rolls to the next lower couple the mixture of shavings which are intermingled as they are disengaged. They are dis-

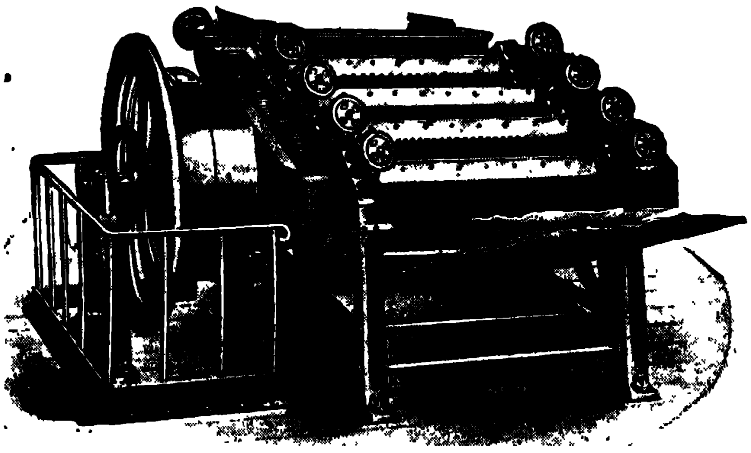


FIG. 154.—Eight-roll Toilet-soap Mill.

charged from the mill at the lowest pair of rolls and fall upon an endless belt which conveys them to the plodder, or they may drop into cars in which they are conveyed to the plodder and from which they are transferred to the hopper of the plodder.

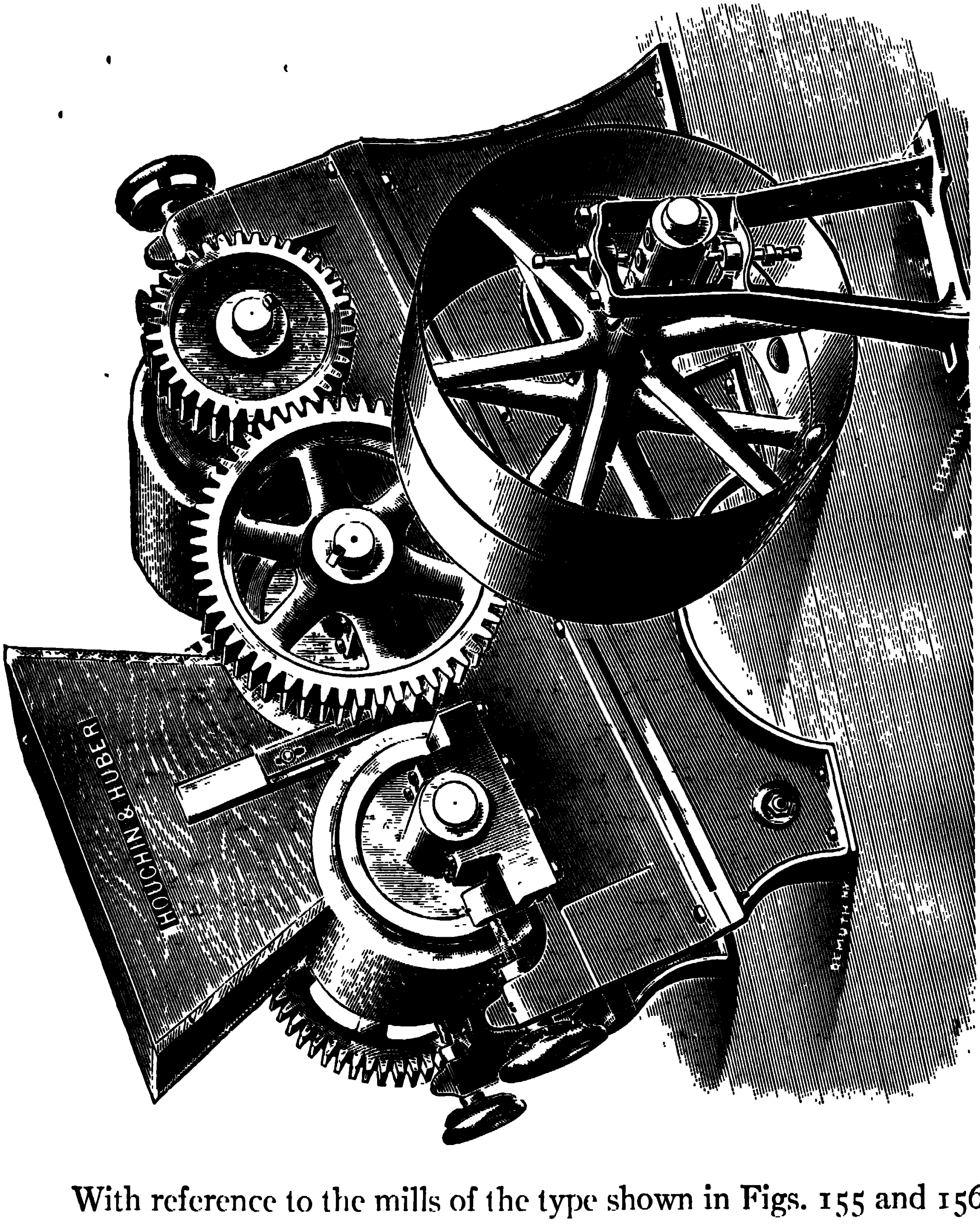


Fig. 155.—Three-roll Mill, 12"×24".

With reference to the mills of the type shown in Figs. 155 and 156, the following practical directions for their adjustment and operation are applicable:

The mill is shipped with the driving-shaft, pulley, and hopper removed to admit of easier handling of the machine. As the mill has been set up and tested at the place of construction it is not advisable to remove the rolls in setting up the mill at the place of use. If found necessary to remove the rolls it should be done by a good

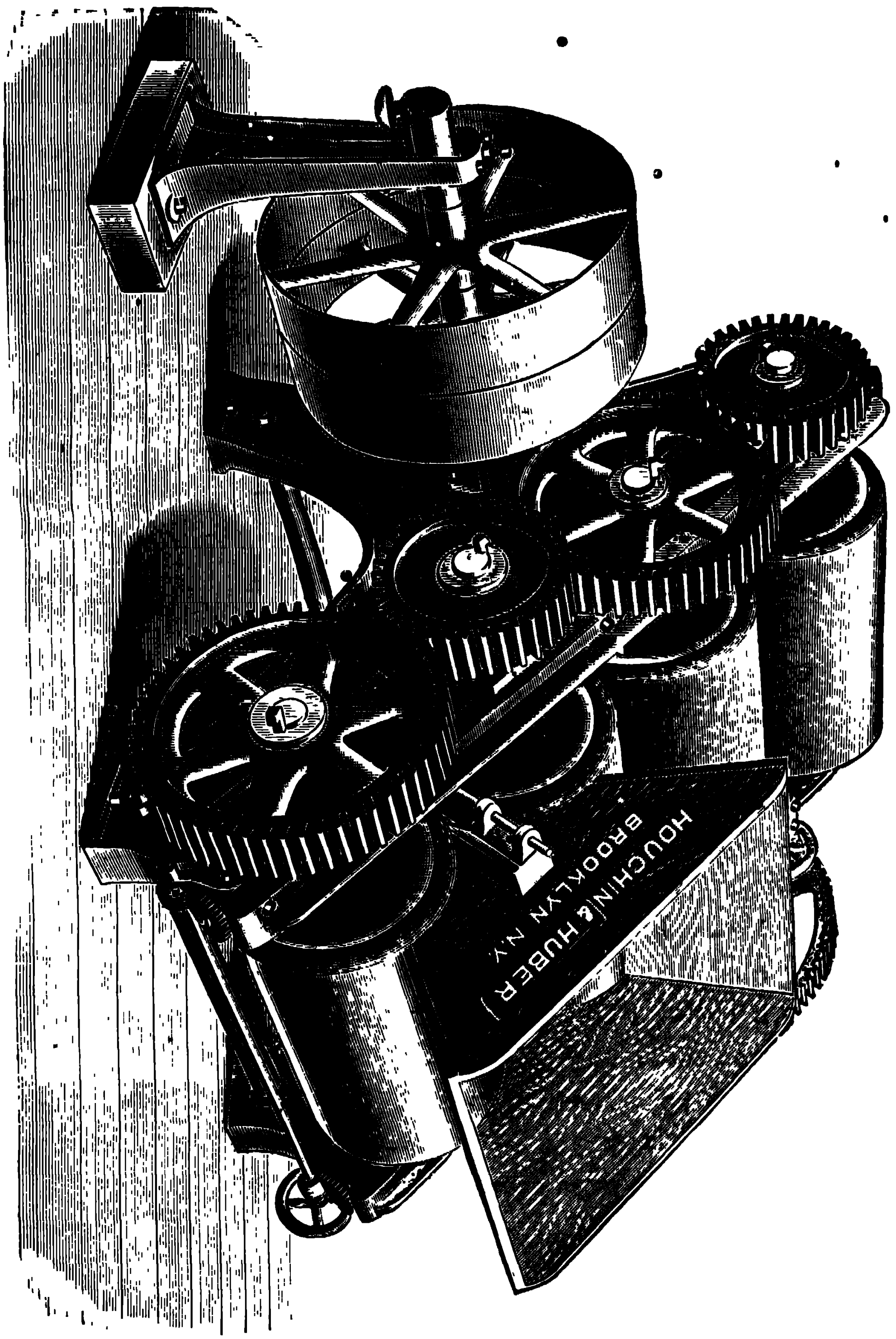


Fig. 156.—Four-roll Mill.

mechanic and the adjustment must be made again by experiment. The adjustment of the mill is simplified by observing the figures from 1 to 5 cut on the small worm-wheels controlling the pressure. In testing the mill the worm-wheels have been set with the figure 2 nearest the centre of the worms and in disturbing the adjustment the operator can always tell that he is nearly right when his adjustment is nearest the figure mentioned. If the number of turns of the hand-wheel are counted when the pressure is relieved, the same number of turns given to the hand-wheel when brought back will secure the exact adjustment each time.

The output of any mill depends to a great degree on the way the rolls are set, and this adjustment will vary somewhat with each grade of soap milled. The best results can be obtained only by experience, which with the method of adjustment as described is soon acquired. Should the rolls wear unevenly in the course of time, the set-screw on the respective worm-gear may be loosened on that side of the mill in which the least wear is shown and the worm allowed to revolve until the pressure is equalized. The set-screw on the loose gear is then adjusted so that both will operate uniformly and secure an equal adjustment throughout the length of the roll in question. To secure the best results the soap must be milled a number of times—as a rule from four to six times. This, however, is determined by the grade of soap and the results desired. The speed of the pulleys should be at least 120 revolutions per minute, although with soap of inferior quality this speed may be increased. Practical work with mills of the type in question has demonstrated that a speed producing more than 1500 inches of surface speed per minute to the top roll is detrimental to a good quality of soap. This applies to large or small rolls, and while it will not harm the mill to use a faster speed, the power required seems to increase out of all proportions to the results obtained and a higher speed is not, therefore, recommended. This factor, however, may be left entirely to the judgment of the operator.

In using one toilet-soap mill in connection with another on the tandem principle, as shown in Fig. 157, it is, as a rule, best to have the first mill revolve a little more slowly than the second in order to avoid the overcrowding of the second mill. This method is made

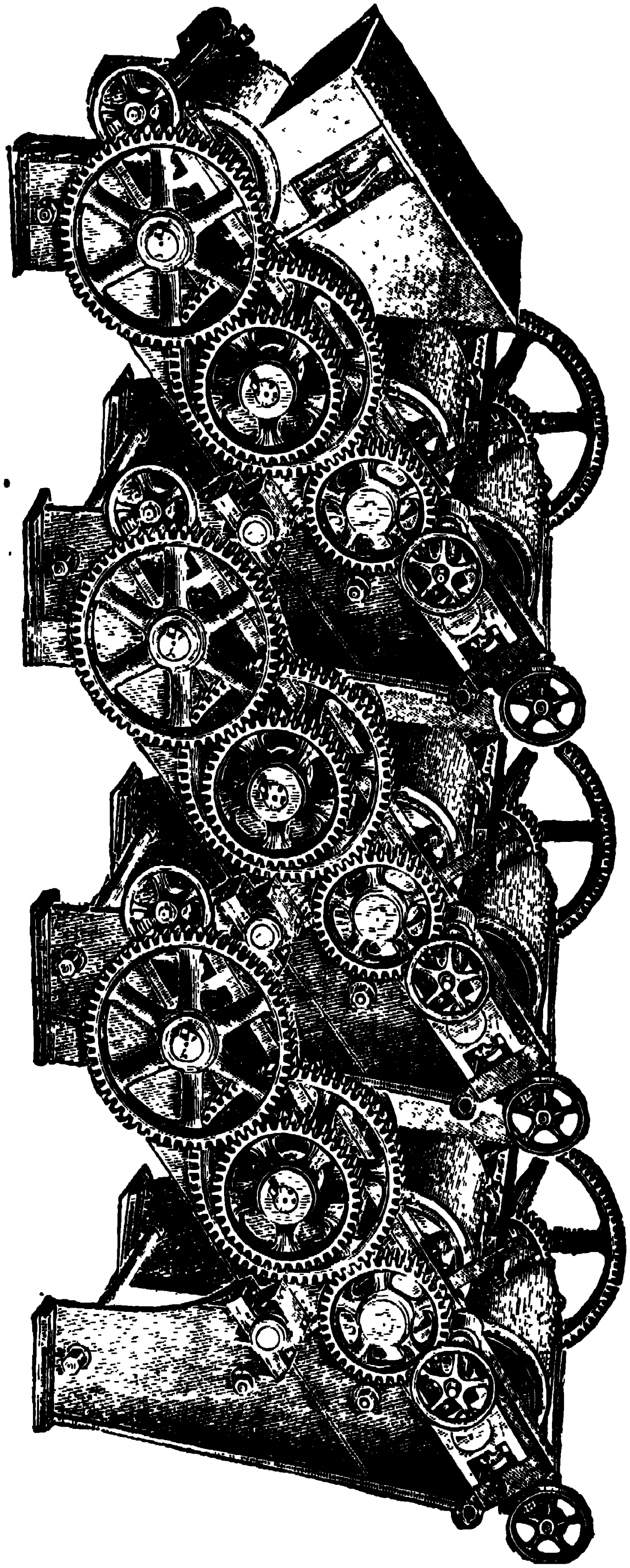


FIG. 157.—Four-roll Mills Arranged in Tandem.

advisable from the increase in the bulk of the soap as a result of milling. It will also be found necessary to study the adjustment of two or more mills working in tandem a little more closely than when the mills are used alone. No definite directions can be given on this point, as the adjustment and operation of the mills are determined by the character of the soap milled. In no case should a mill be allowed to run without soap in the hopper; the friction of rolls upon each other in an empty machine is detrimental to the efficiency of the rolls.

Plodders.—The plodder consists essentially of suitable gearing for the application of power to one or two screws enclosed in a case in which compression of the soap takes place and from which after compression into a compact, homogeneous mass the soap is discharged, in one continuous bar corresponding in cross-section to that of the die, through a nozzle to which is attached a die of any desired cross-section. Sections of the continuous bar are removed as fast as ejected and cut by wire into individual cakes by use of the device shown in Fig. 163.

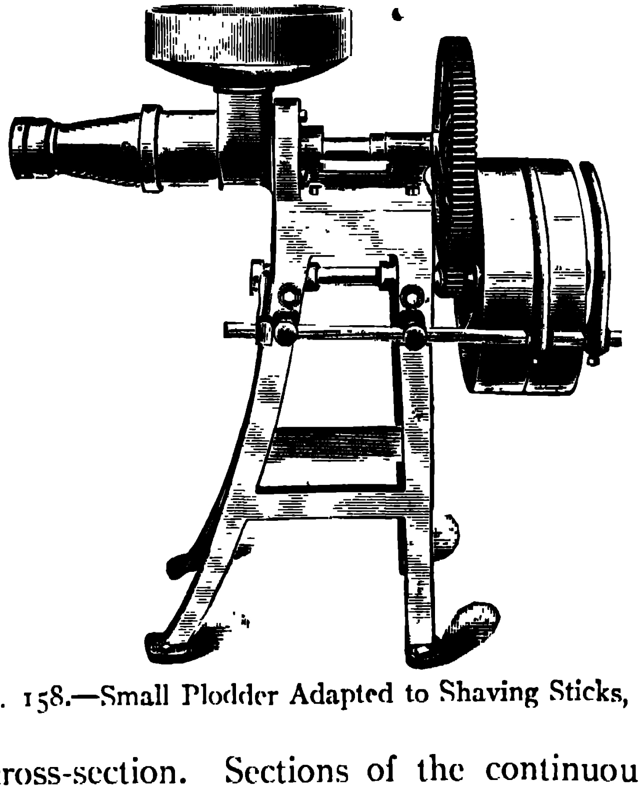


FIG. 158.—Small Plodder Adapted to Shaving Sticks, etc.

The interior construction of single- and double-screw plodders is shown in Fig. 149 and Fig. 150 respectively. In the résumé of

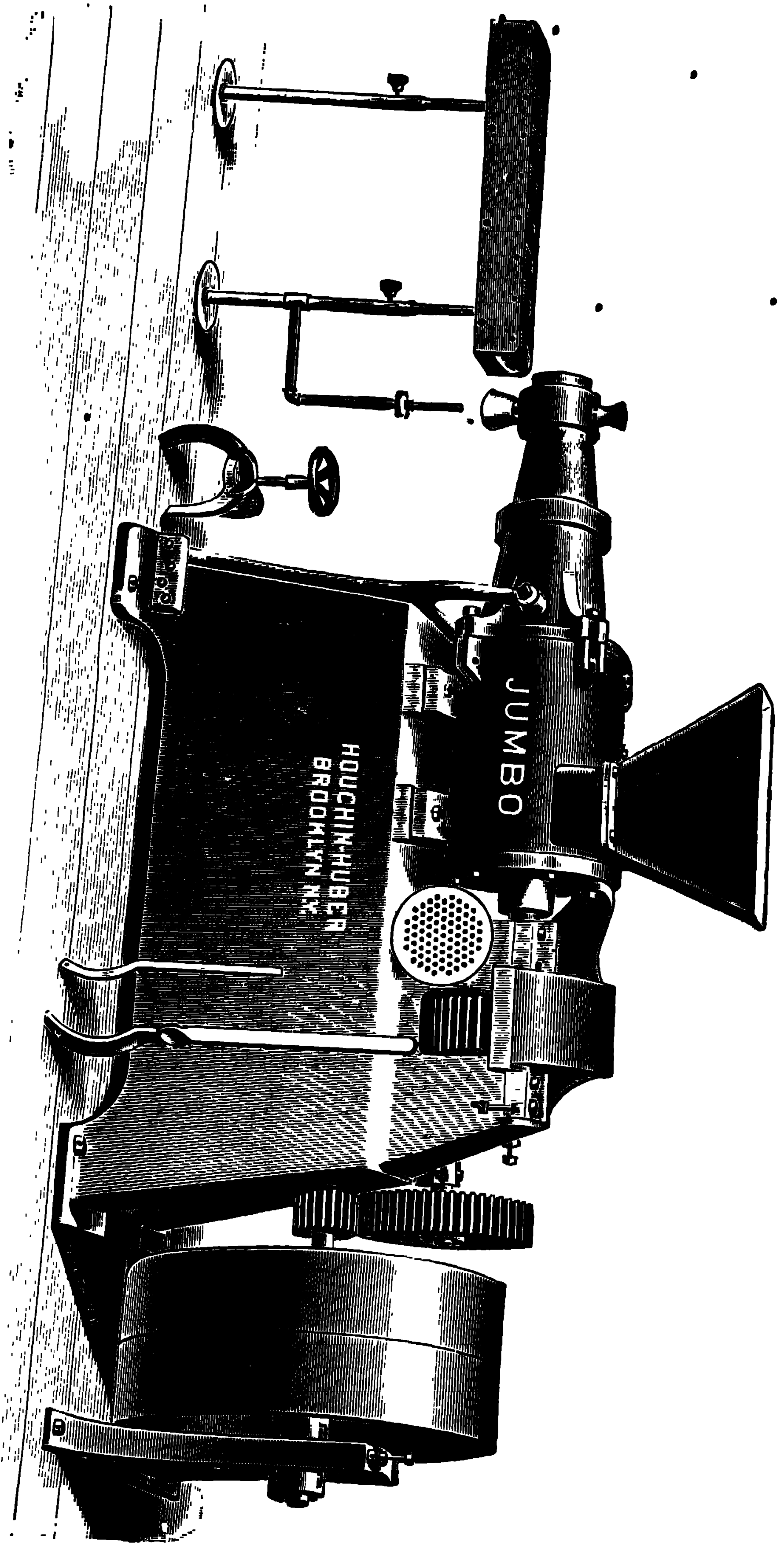


Fig. 159.—Improved Porcelain-lined Plodder with Roller Conveyor.

the growth of milled-soap manufacture it was seen that with the introduction of a plodder providing high compression with large capacity this branch of soap-manufacture made rapid progress. Plodders differ essentially only in one particular, viz., the use of a double or single screw as has been indicated. In Figs. 158 and 159 are shown two plodders of respectively small and large capacity, both of the single-screw type. The application of power by belt and its transference to the screw by suitable gearing and the hopper surmounting the case enclosing the screw are plainly shown. By means of a trunnion fork supporting the cone which is securely attached to the screw-case, the cone may be quickly detached for cleaning the interior of the screw-case.

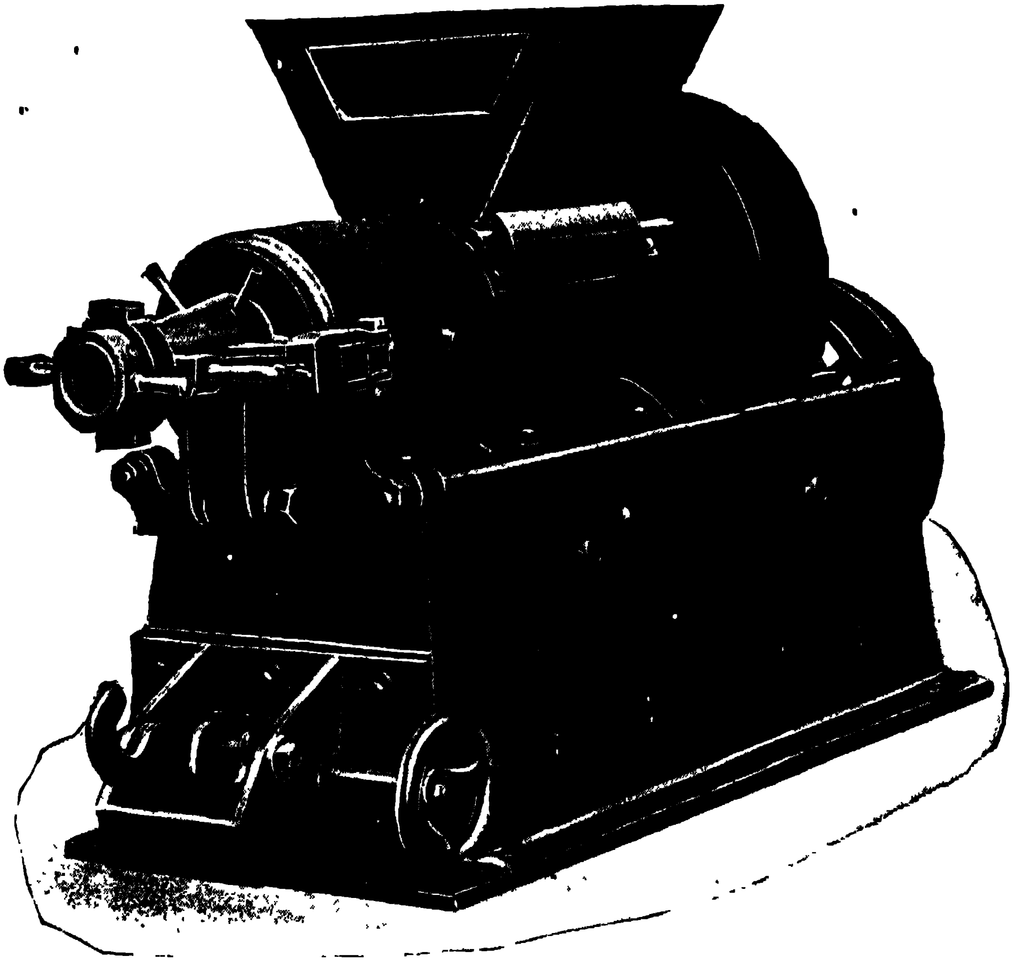


FIG. 160.—Self-feeding Plodder shown in Working Order.

The type of plodder shown in Figs. 160 and 161 is characterized by a patented self-feeding roll which compresses the soap on the revolving screw, and by heating arrangements in the plodder-head whereby compression and homogeneity of the soap are greatly increased. The pipes projecting to the right and left from the upper part of the plodder-head are for the escape of hot air from the chamber in the perforated disc. Heat is applied to the lower part of the plodder-head, in which the disc is located, by means of a Bunsen burner. By the softening of the soap thus effected it is permitted to pass through the perforations of the double-walled disc without being broken by successive revolutions of the screw. A water-chamber is provided as shown at the extremity of the plodder-head. To eject the soap remaining in the plodder-head after each milling, the head is unloosened and lowered as shown and held securely by the hooks. By means of the hand-wheel at the side a plunger thrusts out the soap.

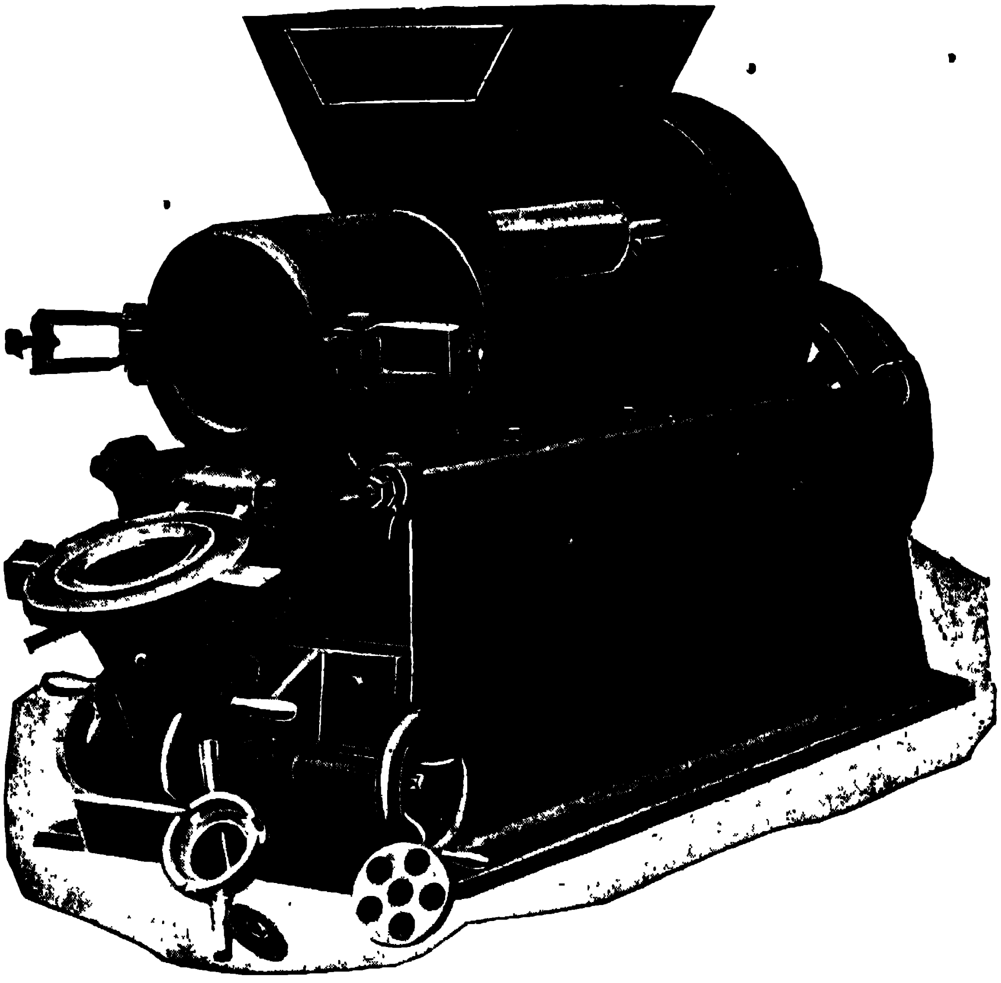


FIG. 161.—Self-feeding Plodder shown with Head Down for Cleaning.

Operation of Plodder.—As shown in the view of the milled-soap plant, Fig. 162, the soap as it comes from the mill in long translucent ribbons of the thinness of paper is transferred directly to the hopper of the plodder, from which, under the action of the

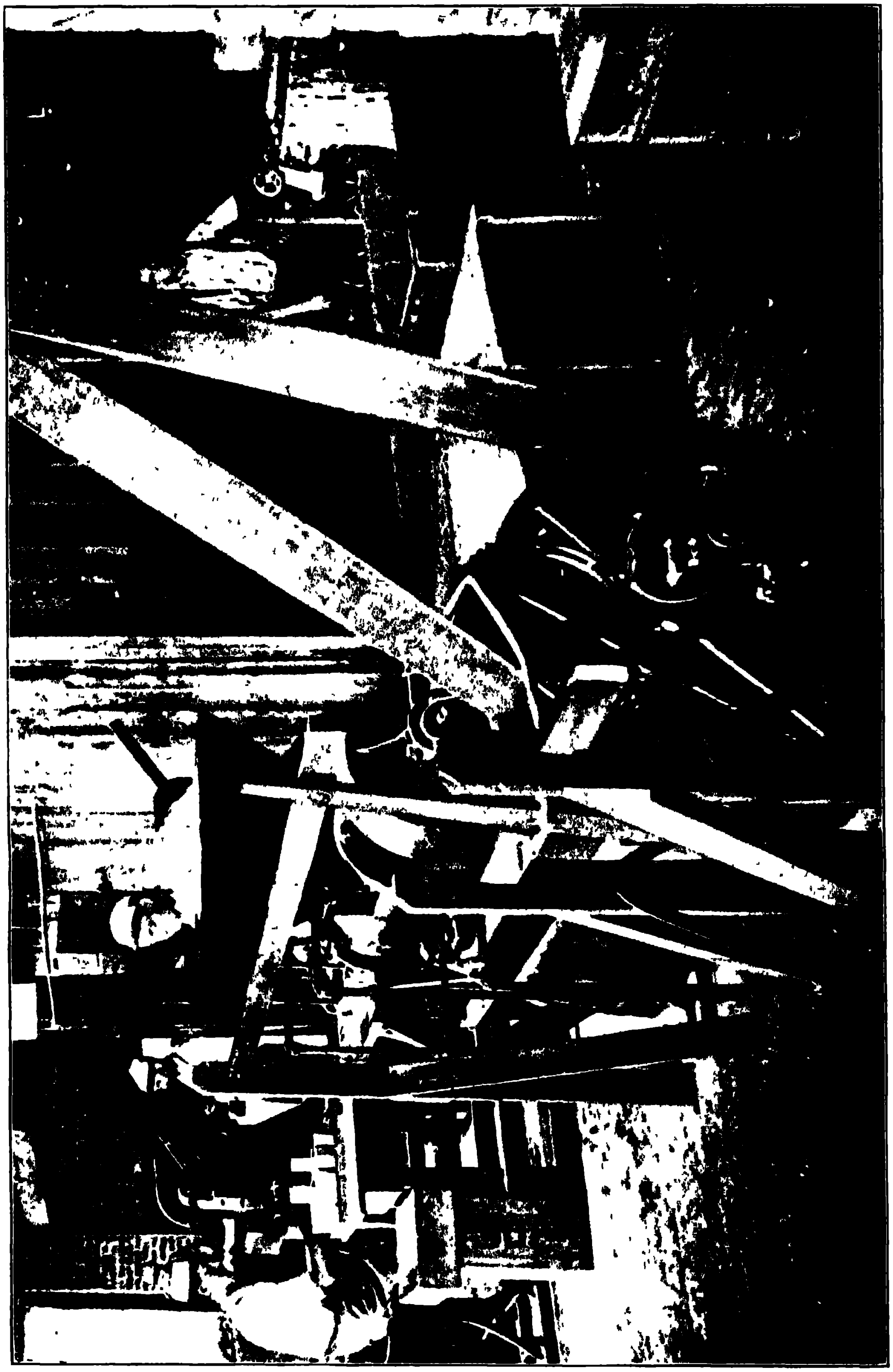


Fig. 162.—View of a Milled-soap Plant.

compressive force of the screw, it is discharged in a compact, continuous bar which is cut into sections and which is again reduced to cakes of the dimensions required by pressing. The character of the product obtained at this stage determines the advisability of any procedure that has preceded. If the soap is too dry after milling, or contains an excessive amount of inert matter, as starch or talc, it tends to crack or "splinter" as it is discharged from the nozzle; such soap is returned to the plodder to be reworked. Cracking at the nozzle, due to friction of the soap against the sides, is increased not only by any treatment that tends to destroy the cohesion of the soap, as the addition of filling material, but is more pronounced with dry than with more hydrated soap and more with soap made from firm than with that made from soft stock. Friction is reduced by softening the soap as it is discharged by means of a hot-water jacket surrounding the nozzle (Fig. 159). The degree of heat should be carefully regulated, as the appearance of the soap is affected by the degree of softening. With suitable heating, if any is required, the soap will emerge compact and smooth; with excessive heating the discharged soap will be streaked after cooling.

In the operation of the plodder (Fig. 159) the following directions apply: The jacket covering the nozzle of the plodder must be filled with warm water or heated with the Bunsen burner. This water jacket must be just hot enough to permit of bearing the hand on it; if too hot the soap will blister. When this occurs the burner can be removed or the flame turned down until the soap comes out in proper condition. Heat may then be reapplied gradually. The cylinder of the plodder is jacketed and cold water should be piped to the bottom of the cylinder and permitted to overflow from the top into a funnel attached to a pipe connected with the sewer; and while in many cases cold water may not be needed, some soaps, where the heat generated by compression is considerable, do require it. Care should be observed to provide for the drainage of the cylinder, otherwise in freezing weather the cylinder may be ruptured.

By reason of the porcelain lining in the plodder it will be found that the compression efficiency is far greater than in plodders of earlier construction. This is due to the fact that soap gets its compression by means of the conveyor feeding the soap to the compression chamber faster than the outlet at the end of this chamber permits ejection. Consequently the more soap that is fed to the compression chamber, and the least resistance it meets with in being conveyed, the more the compression and the less the power required to operate the plodder.

By the smooth interior surface provided by the porcelain lining, adhesion of the soap to the cylinder-wall is reduced to a minimum, which is of great advantage when cleaning the machine or removal of the "plugs," which is oftentimes troublesome when the interior is made of iron or steel.

As the porcelain lining, therefore, has a specific purpose besides giving the interior of the machine an absolute freedom from rust, iron dust, or other annoyances, it is to the interest of the operator to be careful in handling the machine, as porcelain can be chipped if hit hard enough with an iron or steel tool, such as a monkey-wrench, hammer, or any tool whose density is greater than the porcelain itself. Therefore it is advisable to use wooden shovels in feeding the plodder, as well as all mills, since a mill can be broken by having an iron shovel pass between the rolls.

In changing colors, or in cleaning the plodder (Fig. 159), or in changing the die which controls the shape of the elongated, ejected bar, the following particulars are to be noted:

The front cap containing the die is removed first by means of a special wrench. This part of the compression chamber also contains the device for the introduction of water and for the application of heat and is detachable from the rear part of the compression chamber, the two sections being separated at the place of union by a removable perforated screen. These screens, usually two in number, have perforations of $\frac{3}{8}$ and $\frac{5}{8}$ of an inch in diameter respectively. For high-grade soap the screen having the smaller perforations and for low-grade soap the screen with the larger perforations is recommended. It is not advisable to omit the use of the screen altogether; its use, however, is a matter of detail depending upon the character of the soap plodded.

After the removal of the front cap, the next part to be removed is the front part of the compression chamber. It is first given a turn with a large wrench provided for that purpose and the water allowed to flow from the jacket into a receptacle. It is then removed entirely. It will probably be found that the screen or perforated plate adheres to it; if not it may be detached readily by means of a knife. After the removal of the front part of the compression chamber as described, the plodder should be run until soap ceases to be ejected from the rear part of the compression chamber, which operation requires but a few minutes and serves to clean the screw-cylinder completely. The rear part of the compression chamber, which is attached by means of bolts to the cylinder, is next removed by loosening the bolts and allowing it to swing clear from the cylinder by means of the trunnion to which it is attached and which holds it in a stationary position preparatory to cleaning. The plodder is then started slowly and the soap still surrounding the screw will be forced out in a spiral form and may be collected in a box kept for that purpose. To eject the soap the contrivance shown beneath the nozzle of the plodder in Fig. 159 is used. It is held securely against the small end of the rear part of the compression chamber and by very slight pressure produced by turning the screw the plug of soap is forced up far enough to be removed by hand. To remove the plug of soap from the front part of the compression chamber, this part should be placed in an upright position upon two blocks of wood and the plug-ejector attached as before, with this difference, that a small block of wood should be interposed between the screw and the soap. A few turns of the screw will loosen the soap so that the section of the compression chamber may be lifted up and the plug of soap remains in an upright position on the table.

If the removal of the conveyor screw is desired, the nut holding it on the shaft is removed and then a block of wood 2 inches square and about 30 inches long is placed in the hopper to reach the bottom of the cylinder. By turning the machine backwards half a turn, this block of wood will loosen the screw sufficiently so that it can be drawn out by hand, and in placing it on the floor or a table a little care should be taken not to drop it, as that will have a tendency to injure the porcelain. The screw can be pushed back into position very readily and fastened again by means of the nut. The plodder is prepared for use again by replacing the sectional parts in position in the reverse order of their removal.

It is advisable when using different colors to begin with the lightest first, since the plodder will not require washing as when changes from dark brown or tar are made to lighter colored soaps.

It is always advisable where tar soaps are made to have an extra plodder for this work alone, as the odor from tar is difficult to remove from a machine, even should every discernible particle be eliminated.

Should the soap come from the plodder streaky, it is probably due to the fact that the water-jacket of the front nose is too cold; if blistered and soft, too hot.

The Bunsen burner can be regulated to keep the jacket at just the right temperature, and will not require much watching when once understood.

To give the best results in plodding, the soap must have a long grain, and anything done to shorten this grain, as by the incorporation of filling or improper manipulation in the kettle, will cause trouble.

In using the plodder continuously care should be observed to have all bearings well lubricated. The oil-well at the end of the screw-shaft should be kept full of oil, and occasionally this oil must be removed and new oil substituted.

The bearing immediately behind the cylinder should be occasionally oiled with an oil-can, as this is so arranged that oil is not held in this bearing, lest it should find its way into the cylinder.

Pressing.—If in suitable condition the soap may be pressed at once; if too warm or soft it may be necessary to allow the soap to cool and dry. The greater compactness of milled soap demands dies of special construction, which have been previously described. As the shape of the pressed cakes departs more from a square cross-section, the greater compression at the ends of the cakes in pressing shows in the characteristic markings of the finished product. A cake-cutter is shown in Fig. 163.

Continuous Method.—A practical combination of the successive processes of framing, slabbing, cutting, drying, and chipping intervening, the crutcher and the mill may be effected by use of the device shown in Fig. 164. Its operation consists essentially in the application of the fluid soap, to which coloring-matter has been added by crutching, to a hopper surmounting a series of water-cooled cylinders from which the soap as it cools and solidifies is detached by knives in the form of thin shavings. These fall upon a series of endless belts of wire cloth enclosed in a case, suitably heated and ventilated, whereby the soap, reduced to a form that admits of large drying surface, as it is conveyed from one end of the heated chamber to the other and dropped upon successive underlying and moving belts, is discharged with its water content reduced to 12-15 per cent and in suitable condition for milling. Whereas at least seven days are required by methods generally employed, by use of this device the time is reduced to ten to fifteen minutes. The present general use of this device is confined to soap intended for toilet use, but the product made by it is applicable for all purposes. This machine is exceedingly well adapted to the manufacture of "chip" soap on a large scale. The

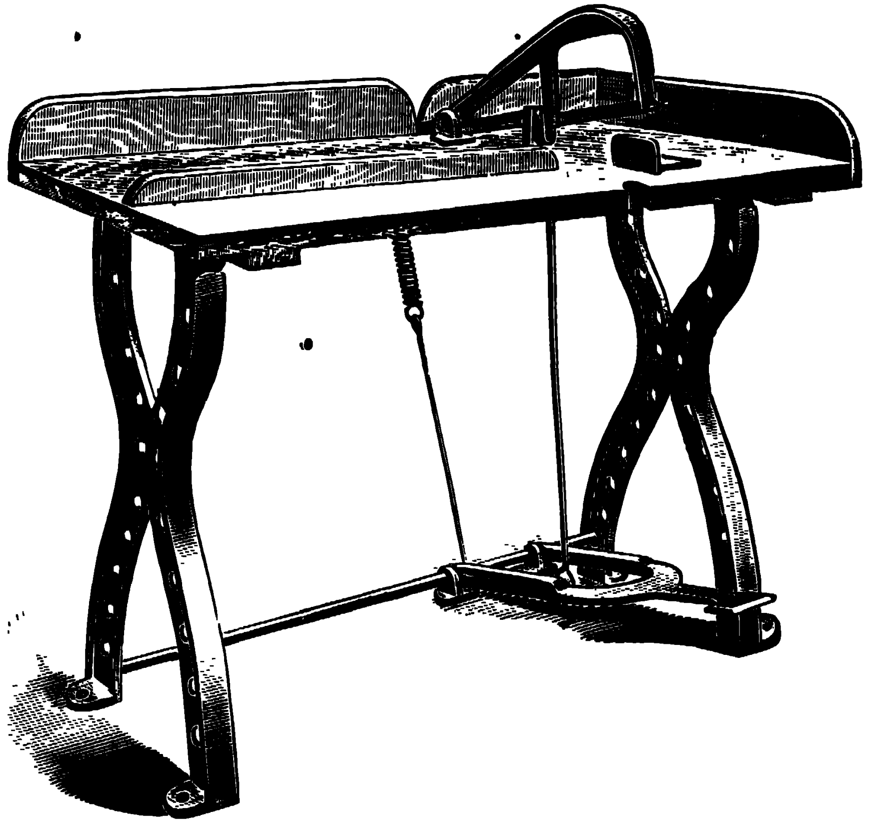


FIG. 163.—Cake-cutter.

product as discharged from the machine is ready for barrelling at once. By suitable regulation of the temperature within the drying chamber the moisture content of the soap may be obtained as desired.

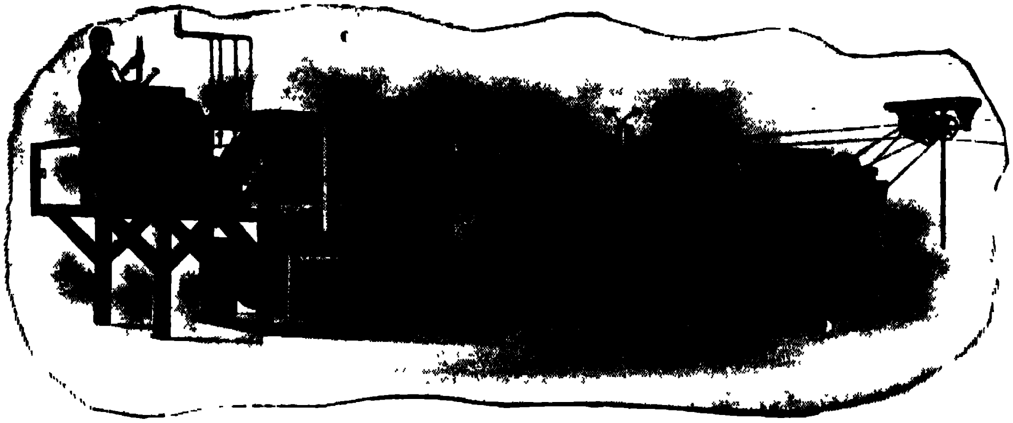


FIG. 164.—Automatic Continuous Crusher and Dryer.

CHAPTER XIII.

CANDLES.

Historical. Raw Material of Fatty-acid Manufacture. Saponification or Hydrolysis. Lime-saponification Process. Use of Process. Construction of Digester. Operation. Treatment of Sweet-water. Treatment of Fatty Acids. Graining or Seeding. Cold-pressing. Hot-pressing. Utilization of Fatty Acids. Acid-saponification Process. Use of Process. Acidification. Construction of Still. Distillation of Fatty Acids. Products of Distillation. Utilization of Products. Manufacture of Hydroxy-stearic Acid. Twitchell Process. Sulpho-aromatic Fatty Acids. Outline of the Twitchell Process. Application of the Process on an Experimental Scale. Preliminary Acid Boil. The Saponification. Further Considerations. Factory Installations of the Twitchell Process. Summary. Paraffin. Stearin. Stearic Acid. Mixtures of Stearic Acid and Paraffin. Miscellaneous Candle-stock. Bleaching Beeswax and "Stearine." The Candle. Preparation of Wick. The Wick. Equipment of the Candle-plant. Melting-kettle. Mixing Ingredients. Coloring Candles. Candle-moulding Machine. Construction. Setting Up and Wicking Candle-machines. Moulding. Operation of Machine. Candle-makers' Tools. Sizes of Candle-machines. Bleaching and Polishing Candles. Classification of Candles. Self-fitting Ends Operation of Self-fitting-end Machines Dipping. Unit of Light Method of Determining the Melting-point of Spermaceti

Historical.—Until Chevreul had begun his scientific investigations in 1811, oils and fats had been regarded as simple organic substances. On the complete publication of his discoveries in 1823, the complex character of these bodies became generally known. Chevreul demonstrated that fats are compounds of glycerin with a fatty body having slight acid properties, called fatty acids, and that when fats are deprived of glycerin, the fatty acids could be rapidly and perfectly pressed and made excellent material for candles. He patented a process for obtaining fatty acids, the process consisting of two distinct stages as follows:

1. Transformation of fat into soap by the use of lime or other alkali; 14 pounds of lime were used to 100 pounds of fat.

2. Decomposition of the soap so produced into fatty acids by the use of 2 pounds of sulphuric acid to each pound of lime.

In association with Gay-Lussac (in 1825) Chevreul attempted the industrial application of the scientific principles he had made known. He did not, however, attain the success he had doubtless anticipated, and it was reserved to M. de Milly to lay the foundation of stearic candle manufacture in 1832. The material operated upon was always tallow, and the process was that of saponification, by lime or by alkalis.

About 1816 Braconnot, a French chemist, discovered that fats are mechanical mixtures, in varying proportions of solid fats, now called stearin, with a liquid fat or oil. He found that simple pressure very slowly applied separated the fluid part and that the remainder made hard candles, but the processs as employed by him was difficult and imperfect.

In 1829 James Soames, of London, patented a plan of separating cocoanut-oil into its solid and liquid constituents by pressure, which was soon brought into use by E. Price & Co. for the production of cocoanut candles and lamp-oil, which firm also introduced an improvement in the substitution of mats made of cocoanut fibre for the canvas which up to that time had been used in the pressing of fats.

In 1831 candle-manufacture in England was set free from the Excise supervision to which it had previously been subjected. From that date its progress was rapid.

The manufacture of the plaited wick, which had been patented in France in 1825 by Cambacères, was introduced into England in 1836. In 1840 J. P. Wilson produced a candle from equal parts of stearic acid and cocoanut stearin in which the plaited wick was used, and which marked an important step in advance. They were named "Composite," because of the mixture of materials in them, but the name has long since passed into a common one for cheap, self-snuffing candles. Palm-oil, treated by Chevreul's process of lime saponification, was brought into limited use for candle-making in 1836, but the dark color of the candles produced limited their general use.

In 1840 a patent was granted to George Gwynne for a mode of effecting the distillation of the fatty acids *in vacuo.* This, as the first attempt to apply commercially the knowledge that the acids could be so distilled without decomposition, was an important step; but it was superseded, in 1842 and 1843, by a discovery patented by E. Price & Co. in the names of William C. Jones and George F. Wilson. The patentees found that all the good effects of the vacuum-apparatus could be gained by the use (in the distillation of fatty acids) of free steam, and that the costly and complicated vacuum-still could be replaced by an apparatus very simple in construction and easily managed while in operation. And further, the inventors pointed out that fats might be acidified in an advantageous manner by treatment with sulphuric acid, if only the proper conditions were assured.

Frèmy had made known in 1836 that fats become greatly hardened by being treated with *half their weight* of sulphuric acid, care being taken to keep the mixture cool by means of a freezing compound. This process would be wholly impracticable in practice. Frèmy explained the decomposition of fats by sulphuric acid by the formation of unstable sulphonated compounds of fatty acids and glycerin, which on subsequent boiling with water resolved themselves into sulphuric acid, fatty acids, and glycerin.

Jones & Wilson showed that if the fat were *not cooled, but heated,* then 33 per cent of acid would produce all the good results noted by Frèmy. This process came at once into practical use. Clark & Gwynne had patented in 1840 the use of strong sulphuric acid in treating fats and oils; but both patentees described the addition of the acid to a cold or nearly cold material.

Perhaps no discoveries have had so great an influence on candle-manufacture as those which were embodied in Jones & Wilson's patents of this period. The decomposition of fat by means of alkalis or lime had been up to this time the only process known for the production of fatty acids. But this process effected only a small change in the color or odor of many dark-colored and offensive fats; and although by the pressing of such fats, after the separation of the glycerin, much of the color and of the odorous principles are removed with the liquid fatty acid, yet it is rarely that a material so white and so free from smell as the public require their candles to be can be procured by the alkaline-saponification process from dark-colored or offensive fats. But with the power of producing colorless hard acids by means of sulphuric-acid treatment, distillation, and pressing, the candle-manufacturer found his range of raw materials immensely increased. Up to the time of these inventions tallow was the only material upon which he could work freely; and even of this costly material the lower qualities were unfit for his use. But with Jones & Wilson's inventions he could fearlessly operate upon bone and skin fats, fish-oils, greases recovered from other processes, and, above all, upon palm-oil, and from each of them obtain a white and inodorous material for his candles.

Improvements in various directions were made upon Jones & Wilson's inventions: especially the proportion of acid has been reduced from 33 per cent to 10, 6, 4, and even 3.5 per cent by suitable modification of other conditions. The distillation process was introduced into France in 1846. By 1853 oleic acid (red oil) was generally introduced for the oiling of wood both on the Continent and in England. Heretofore, owing to contamination with mineral acid used for decomposition in the saponification process, oleic acid, forming, roughly, 50 per cent of the total fatty acids, had been salable only at an unsatisfactory price.

In 1853 R. A. Tilghman discovered that fatty acids could be separated from glycerin, without injury to the latter, by the single and simple process of subjecting the neutral fat while in intimate mixture with water to a high degree of heat, under sufficient pressure to prevent the water from being converted into steam, without the employment of any alkali or sulphuric acid or other saponifying agent, the operation, even with the most solid fats, being capable of completion in a very few minutes when the heat applied is equal to that of melting lead, or 612° Fahr., but requiring several hours when it is as low as 350° or 400° Fahr. The only conditions are a constant and intimate mixture of the fat with the water, a high degree of heat, and a pressure sufficiently powerful to resist the conversion of the water into steam. The result was a decomposition of the fatty body into its elements of glycerin and fatty acids, each element taking up the requisite equivalent of water essential to its separate existence, and the glycerin in solution separating itself from the fatty acids by settling to the bottom, when the mixed products are allowed to stand and cool. In this process a chemical change took place in the fat in consequence of the presence of the water and the active influence of the heat and pressure upon the mixture. In 1854 Tilghman patented his discovery in England and in the United States.

However, owing to the incomplete saponification and the destruction of the fatty acids, the industrial application of this process was not successful.

Chevreul's lime-saponification process and the sulphuric-acid distillation process were the only two processes practically used down to the time of Tilghman's invention.

G. F. Wilson, of Price's Patent Candle Co., Ltd., in a paper contributed to the *Journal of the Society of Arts*, Jan. 5, 1856, states that the process of Tilghman suggested to them the idea of distilling fats by passing steam into them at a high temperature, whereby to resolve them into glycerin and fatty acids. They found the plan successful, and that the glycerin distilled over with the fatty acids, but no longer combined with them, and in July, 1854, they took out a patent for that purpose. In a paper read before the Glasgow meeting of the British Association for the Advancement of Science, in September, 1855, Mr. Wilson thus refers to the course of discovery which took place in this branch of manufacture:

"By our first improvement in separating the fatty acids from neutral fats the glycerin was decomposed by the direct action of concentrated sulphuric acid at a high temperature, and all that remained of it was a charred precipitate. A new process for decomposing neutral fats by water under great pressure, coming under our notice, referring to Tilghman's process, led us to look again more closely into our old distilling processes, and the doing this showed, what we had often been on the brink of discovering, that glycerin might be distilled.

"In our new process the only chemical agents employed for decomposing the neutral fat and separating its glycerin are steam and heat, and the only agents used in purifying the glycerin thus obtained are heat and steam. Thus all trouble from earthy salts or lead is escaped. Distillation, however, purifies the impure glycerin of the old sources."

The discovery in England in 1855 by G. F. Wilson and G. Payne, as before stated, contributed to the restriction and final abandonment of Tilghman's process in that country. This discovery, which was patented, was, viz., that neutral fats could be, by distillation with superheated steam alone, broken up into fatty acids and glycerin. Before the close of the same year G. F. Wilson discovered that glycerin could be distilled without decomposition by the use of steam. Previous to this date a chemically pure glycerin had never been seen. Such a material became possible under this new patented discovery.

In 1850 James Young patented a process for the production of hydrocarbons by the distillation of coal at a low red heat. Oil of the same chemical composition had for centuries been known to exist in abundance in Burmah, and had been described in scientific books under the name of petroleum, or rock-oil; but because of its impurity it had been used only for rough purposes in the East, without becoming an article of importation, to any large extent, into Europe. In 1853 and in the following years patents were granted to Warren De la Rue for the working of this Burmese, or Rangoon, petroleum. The utilization of this material acquired commercial importance and in the first half of 1859 burning oil obtained therefrom was exported to New York from England to the extent of 10,600 gallons. Before the close of this year, however, the discovery of petroleum in the United States revolutionized the geographical distribution of this material and relegated Rangoon petroleum to commercial obscurity, which prevailed until recent years. Paraffin appeared first as a candle stock in 1850, made by a process patented by Young in that year. The production of this solid hydrocarbon was comparatively small for many years, but it was largely increased when the refiners of American petroleum began to devote their attention to the utilization of the residuum remaining after the separation of the more volatile portions of crude petroleum.

The form of autoclave, or digester, at present in use was introduced by de Milly in 1855; while being practically a modification of Tilghman's digester, it was the outcome of experiments undertaken by de Milly and Motard, as early as 1834, to effect the decomposition of fats under pressure. Tilghman's apparatus, which was constructed of wrought iron lined with lead and was placed horizontally, was worked at a pressure of 250 pounds per square inch, no agent except water being employed for the decomposition of the fat. M. de Milly's autoclave, which was constructed of copper and placed vertically, was worked at a lower pressure, viz., 120 pounds per square inch, but required the employment of a small percentage (2 per cent) of lime, or of an equivalent saponifying agent.

During 1880-1885 the increased production of paraffin, both from American petroleum and Scotch shale, resulted in its general use as candle-stock and a considerable reduction in the price of candles.

The first recorded improvement upon the candle-mould, attributed to Sieur de Brez, of the fifteenth century, was made by Thomas Binns in 1801 and consisted of the alternate application of heat and cold to the moulds enclosed in a tight box, which is the precursor of the mould-box of the present candle-machine. In 1832 William Palmer repatented the mould-box originated thirty years before by Binns.. In 1834 Joseph Morgan patented a moulding-machine which contained the essential elements of the device at present in use. Morgan's machine was continuous-wicking, was provided with hollow movable pistons for expelling the moulded candles and a clamp for holding them when expelled. Palmer made other improvements of greater or less practicability, notably in 1845, when he placed three series of moulds vertically superimposed, whereby he was enabled to wick them together. Each series was connected with hot and cold water and when filled and cooled and the wicks cut, was removed and the candles rammed from the moulds. In 1855 John Stainthorp, an American, combined the practicable elements of preceding designs into a very compact and serviceable machine, which, while incorporating no absolute novelty, made possible efficiency greater than had hitherto been attained. With the exception of moulds for self-fitting ends Stainthorp's ideas are embodied in the machines described on subsequent pages. Humiston, an American of English birth, contemporaneously with Stainthorp attained the same ends, but Stainthorp's American patents superseded his; Humiston's English patents, taken out in 1857, superseded Stainthorp's by but three days. While some modification in cooling and heating the mould-box has been made, together with more substantial construction and increase in capacity, the modern candle-moulding machine exists practically as Stainthorp left it.

Raw Materials of Fatty-acid Manufacture.—Previous to the introduction of a practicable distillation process, the raw material for fatty acids was confined to the better grades of tallow, but with its introduction the field was enlarged to include palm-oil and dark and inferior oils and greases and recovered fatty products, from which by acid distillation directly, or subsequently to lime saponification, white and inodorous fatty acids were possible. Palm oil may be bleached readily by the bichromate sulphuric acid method described on page 127; by the acid distillation process the color and odor are both destroyed, while with alkaline saponification, either in an open kettle or under pressure, both the color and odor persist. The availability of any material is determined by its relative economy with respect to that of tallow or palm oil. The suitability of any fatty material for fatty acids depends upon its yield of stearic acid, which is indicated by its body or firmness and which may be determined accurately by ascertaining the melting-point of the original material, or the titre, or solidifying-point of the purified and dry fatty acids obtained from the material

Saponification, or Hydrolysis.—The term "saponification" is applied generally to those chemical reactions in which the glycerides constituting fats or oils are decomposed whereby fatty acids are liberated and a body with a constitution characteristic of the alcohols is formed. The term also includes the similar decomposition of the waxes which are compounds of fatty acids with mono or diatomic alcohols. The term "hydrolysis" is often used synonymously, the etymology of the term laying stress upon the fact that water is instrumental in effecting the decomposition, though it must be borne in mind that water alone must be considered the decomposing agent whether the term "saponification" or "hydrolysis" be applied to the reaction. More strictly the term "hydrolysis" should apply to those reactions wherein the fatty acids liberated remain in an uncombined state. Water under normal conditions of temperature and pressure, save in the presence of alkalis, acids, or ferments, is without effect in the decomposition of glycerides, but at a temperature of 200° C. (392° Fahr.) and under normal pressure, water decomposes the glyceride with the formation, by the absorption of water, of fatty acids and glycerin. In the presence of concentrated mineral acids, as sulphuric or hydrochloric acids, the decomposition under normal pressure may be effected at temperatures as low as 100°–120° C. (212°–248° Fahr.). Inasmuch as the formation of fatty acids and glycerin occurs only by the absorption of water, it is a condition precedent to the decomposition of the glyceride that water be present. As stated, the decomposition of the glyceride may be hastened under suitable conditions by the presence of alkalis, acids, or ferments. These bodies are termed catalytic agents or catalysts, and the process, catalysis, by which is understood a change by virtue of the mere presence of bodies which do not of themselves enter into the reaction, or if they do in some way enter into the reaction they are in the course of it always brought back to their original condition. Where the catalytic agent is an alkali, as in the instances mentioned before, a further reaction ensues in the combination of the fatty acids with the alkali. With the high temperature accompanying increased pressure water is effectual in decomposing glycerides. In the following table by Klimont * is shown the comparative rate of hydrolysis of different stocks under pressures of 105 and 225 pounds respectively. The percentage decomposition may be indicated roughly by halving the acid values; in the case of cocoanut oil the figure thus obtained should be multiplied by 0.4.

The bases commonly employed as catalytic agents to accelerate

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* Zeit. für angew. Chemie, 1901, 127.

TABLE XXVI.—AQUEOUS SAPONIFICATION OF NEUTRAL FATS UNDER PRESSURE 30 GRAMS OF OIL OR FAT AND 500 GRAMS OF WATER.

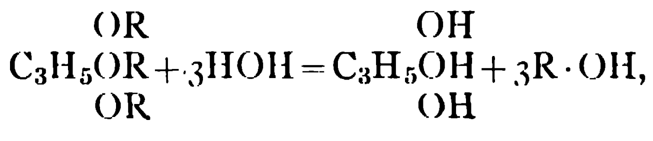
Oil or Fat.	Steam-pressure of 105 Lbs. Acid Values After				Steam-pressure of 225 Lbs. Acid Values After			
	2 Hrs.	4 Hrs.	6 Hrs.	8 Hrs.	1½ Hrs.	2 Hrs.	4 Hrs.	6 Hrs.
Cocoanut-oil	0.1	0.3	0.5	0.9	78.6	90.2	123.9	185.5
Japan wax	1.8	5.3	9.4	13.1	...	12.3	32.5	46.1
Tallow	17.5	37.3	67.3	81.8	....	62.3	106.3	155.8
Pressed tallow	15.3	38.3	65.5	81.6	....	60.4	98.7	160.3
Cocoa-butter	12.3	24.5	45.1	62.6	....	34.5	76.1	160.5
Olive-oil	15.1	32.1	53.0	71.4	....	66.5	114.5	159.5
Sesame-oil	14.3	31.1	56.2	76.0	....	61.7	108.4	153.7
Cottonseed-oil	10.0	23.2	36.3	51.7	....	42.2	80.2	128.6
Linseed-oil	11.4	21.1	43.3	56.1	....	38.1	78.5	130.5

aqueous saponification are caustic soda in soap-manufacture and lime in the manufacture of fatty acids for candle stock. In soap-manufacture this decomposition is effected under atmospheric pressure by boiling the stock with the alkali hydrate caustic soda; Chevreul and Gay-Lussac under similar conditions in the preparation of fatty acids used the alkaline-earth hydrate, slaked lime.

In the consideration of the factors of temperature (pressure) and the proportion of base present we may assume two extremes of conditions, viz., that of atmospheric or normal pressure and an excess of base present, and that of increased pressure and a minimum proportion of base present. In the former condition, as demonstrated orginally by Chevreul, the molecular proportion of the base present is insufficient to effect complete decomposition, he having found that instead of the theoretical amount of 9.7 per cent of CaO, 12-14 per cent were required. The same principles apply to soap-boiling. Inasmuch as the rapidity of decomposition is determined by the quantity of the base present, we find at this extreme the most favorable conditions in point of time, regardless of the state of the product.

In the latter condition, i.e., in the case of hydrolysis under pressure with a minimum proportion of the base present, it is not essential that the base be present in molecular proportions. With increased pressure (temperature) the proportion of the base may be gradually reduced until at a pressure of 180 pounds, corresponding to a temperature of 220° C. (428° Fahr.), 1 per cent of the base is sufficient for practically complete hydrolysis. The time required for hydrolysis is determined, therefore, by the quantity of the base present and the pressure (temperature). With hydrolysis effected in the shortest time, viz., under normal pressure and in presence of an excess of the base, the fatty acids enter into combination with the base to form soap. To obtain the fatty acids in a free state, many hours are required for the hydrolysis of the stock maintained at a high pressure and in intimate admixture with a minimum proportion of the base. At the completion of the reaction the amount of base used is in combination with a proportionate amount of fatty acids and remains, as will be seen later, to be separated by treatment with acid. In accordance with the chemical law of mass-action, to the effect that where one substance is decomposed by another, with the formation of dissimilar substances, the action will grow less and less as the masses of the original substances decrease, and finally a stage will be reached before the decomposition is complete when no further action will take place, or a state of equilibrium will be established between the original substances and the products of their decomposition. We find that the rate of hydrolysis is greatest at the beginning and gradually diminishes until an equilibrium is attained. The stage is represented by the tendency of the base to combine with the liberated acids to be equal to the opposite tendency of the water to dissociate the soap thus formed.

Until recently the equation representing the hydrolysis of oils and fats, i.e.,



$$C_3H_5\begin{matrix}OR\\OR\\OR\end{matrix}+3HOH=C_3H_5\begin{matrix}OH\\OH\\OH\end{matrix}+3R\cdot OH,$$

in which R indicates the fatty-acid radicle, was taken as the true expression of the reaction in the sense that one molecule of triglyceride was converted into one molecule of glycerol and three molecules of fatty acids. Geitel, however, first showed by physico-chemical experiments that the reaction underlying the hydrolysis is a bimolecular one; according to this view the saponification of glycerides takes place in stages, the triglyceride passing through the diglyceride into the monoglyceride, to be finally resolved into glycerol and free fatty acid. As the correctness of this view was doubted Lewkowitsch tried to prove its correctness by purely chemical methods. If diglycerides and monoglycerides did appear as intermediate products in a partially saponified fat, then it should be possible to prove the presence of lower glycerides by converting the lower glycerides into a triglyceride by boiling with acetic anhydride. The then resulting triglycerides could be readily examined for the presence of acetyl groups. On carrying out the experiments, Lewkowitsch actually proved that acetic acid was obtained on saponifying the acetylated mass. The acetic acid could be determined quantitatively in the usual manner. Now if a natural fat was saponified slowly in a manner simulating the process carried on on a large scale, and samples from the partially saponified mass were acetylated, varying amounts of acetic acid were obtained from them. On plotting the results in a system of coördinates, zigzag-like curves were observed. This proved that lower glycerides did occur in the partially saponified mass, and hence that the progress of saponification takes places in accordance with the view that the triglycerides pass through the diglycerides and monoglycerides, and that all three possible reactions occur simultaneously.

Lime-saponification Process.—The original lime-saponification process, as discovered and patented by Chevreul and first applied commercially but unsuccessfully by him and Gay-Lussac in 1825, consisted in the complete saturation of the neutral fat with lime or alkali. In the commercial operation of the process by these two scientists alkali intsead of lime was employed. Chevreul suggested that the production of soap could be expedited by boiling the fat and alkali solution together under pressure. De Milly with greater commercial acumen assumed the enterprise abandoned by Chevreul and Gay-Lussac, and by using the cheaper lime for saponification placed the enterprise upon a sound basis. The stearic-acid candle industry dates from de Milly's efforts in 1832. In 1834 de Milly found that by decomposing fats under pressure in a closed vessel, the amount of lime necessary could be reduced from the amount used, viz., 14 pounds (the theoretical amount is 9.7 pounds) per hundredweight of fat under atmospheric pressure to 4 per cent.

Tilghman's discovery in 1853, that neutral fats could be decomposed at high heat by water alone under pressure in a suitably constructed vessel immediately suggested to de Milly certain improvements in the process which resulted in the apparatus and procedure employed to-day, which is essentially a combination of the original process of Chevreul and the Tilghman process previously described.

Use of Process.—Where used alone without the adjunct of the distillation process, the lime-saponification process is adapted only for stock of superior quality, the product of which may be pressed at once and the solid fatty acids utilized; where used in conjunction with distillation, any stock of suitable firmness is available, the quality of the product of saponification determining its immediate utilization or subsequent distillation. The lime-saponification process yields the maximum percentage of glycerin and fatty acids only in practically the same relative proportions in which they exist in the original material and without deterioration. There is no residue such as characterizes the distillation process. Where glycerin is the object, the autoclave, or lime-saponification process is to be preferred. The term "lime saponification" applied to the process of hydrolysis refers more strictly to Chevreul's original process wherein he completely saturated the fatty acids with lime and later set them free by treatment with mineral acid. It has come to be applied to aqueous saponification under pressure in which lime is employed solely as an accelerator or catalytic agent, and in this significance the term is used here.

Construction of Digester.—The digester, or autoclave, employed in the lime saponification of fats and oils and devised first by the Frenchman, Dr. Papin, in 1690, is similar to the modern rendering-tank in that the active agent is steam under pressure, aided, however, by the addition of from 1 to 3 per cent of lime. The digester is essentially a closed, tightly riveted, and calked cylindrical sheet-steel tank which may or may not be lined with copper or enclose a copper tank of smaller diameter. It may be placed vertically or horizontally, but preferably the former. They vary in dimensions from 3 to 6 feet in diameter and from 18 to 30 feet in height. When made entirely of copper they measure, as a rule, 5 feet in diameter and 18–20 feet high. By use of copper or copper-lined digesters, discolorization of the fatty acids which would otherwise be considerable is reduced to a minimum. The upper cover is made of stronger metal than the rest, and, like the detachable bottom, it is held in its place by bolts and nuts. A manhole is provided either in the top or side of the digester. Suitable openings are provided by means of copper pipes and cocks for the admission of fat, lime-water, and steam, and for discharging the contents to suitable vessels where the separation of the fatty acids and lime soap from the sweet-water and the decomposition of the lime soap into fatty acids are effected. Three-way cocks are used whereby different material may be admitted into and discharged from the digester through the same pipe. The digester is insulated or jacketed with suitable material, either wood or asbestos, to economize heat and therefore fuel, and is provided also with a steam-pressure gauge.

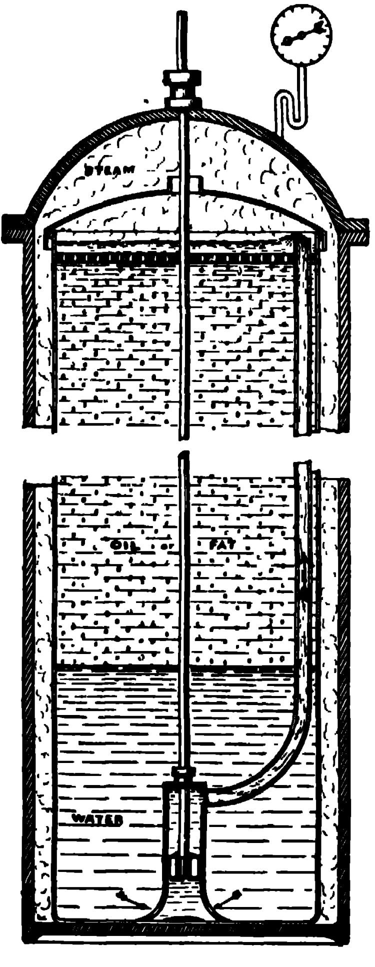


FIG. 165.—Interior Construction of the Digester.

Agitation may be effected in a variety of ways on the same principle as obtains in the ordinary soap-crutcher, viz., by means of paddles spirally inserted on a vertical or horizontal shaft, according as the digester is vertical or horizontal, or by means of a screw enclosed within an inner concentric cylinder. A pump may be used as shown in section in Fig. 165, in which the interior of a digester is shown. The same digester is also shown in Fig. 166, illustrating the cross-section of a digester-house. In Fig. 167 is shown the top of a digester with the location of the pump for the agitation of the contents of the digester and attachments for the admission and discharge of material.

In R. A. Tilghman *versus* Procter *et al.** the following description of the digester shown in Figs. 165, 166 and 167 is given:

"The apparatus consists of a digester, or strong iron tank, of the dimensions of about 25 feet in length and about 4 feet internal diameter. Within said tank is placed a second tank, made of copper, slightly less, say 4 to 6 inches, in diameter than the exterior tank, the interior tank being bolted at its bottom to the exterior tank.

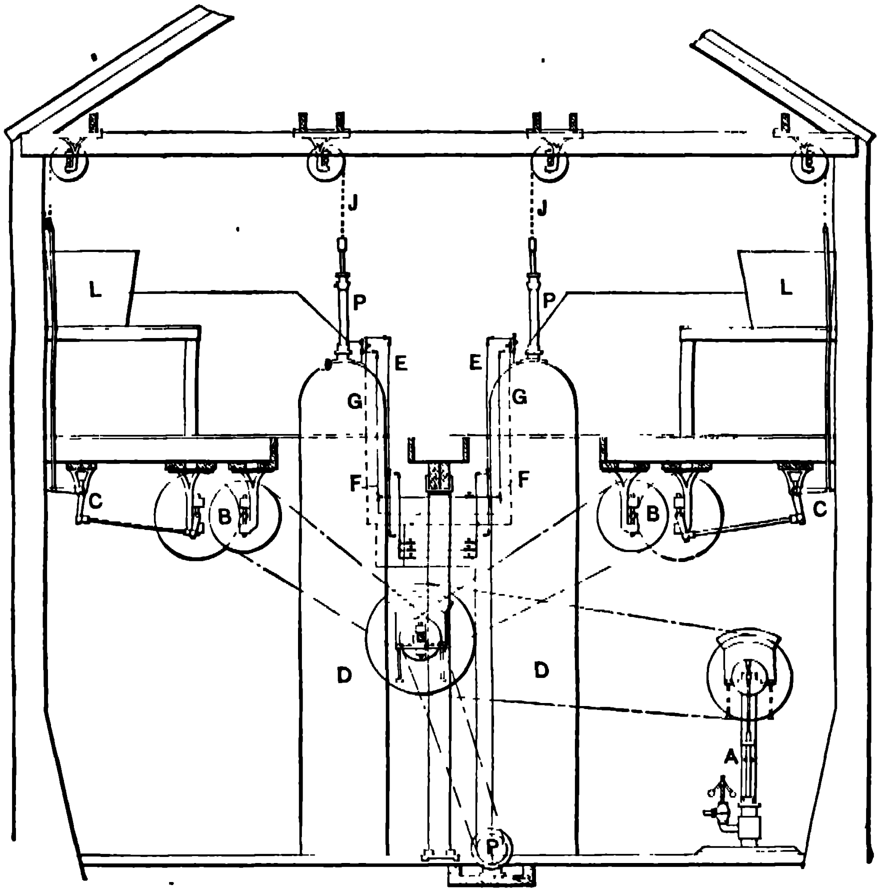


FIG. 166.—Cross-section of Digester-house.

"Into the interior tank there is placed fat and water in equal quantities, together with lime, in quantity equal to 1 per cent of the quantity of fat, the usual charge of fat being 6500 to 7000 pounds, according to the quality and kind employed.

"When the fat, water, and lime have been placed in the digester, or tank, the digester is not full by several feet.

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* See the following reports of the U. S. Supreme Court: Mitchell *v.* Tilghman, 19 Wallace, 287; Tilghman *v.* Procter, 12 Otto, 707.

"Upon the introduction of the fat, water, and lime, superheated steam, at a pressure of between 200 and 250 pounds to the square inch, is introduced near the top of the iron tank, and finds its way, through numerous holes left for the purpose, into the interior copper tank, and is there brought into contact with, and made to operate upon, the contents of the interior tank during the time that the charge of fat remains in the tank, during all of which period steam in large quantities is necessarily and intentionally present in said tank with the fat; that the duration of the operation is from 9 to

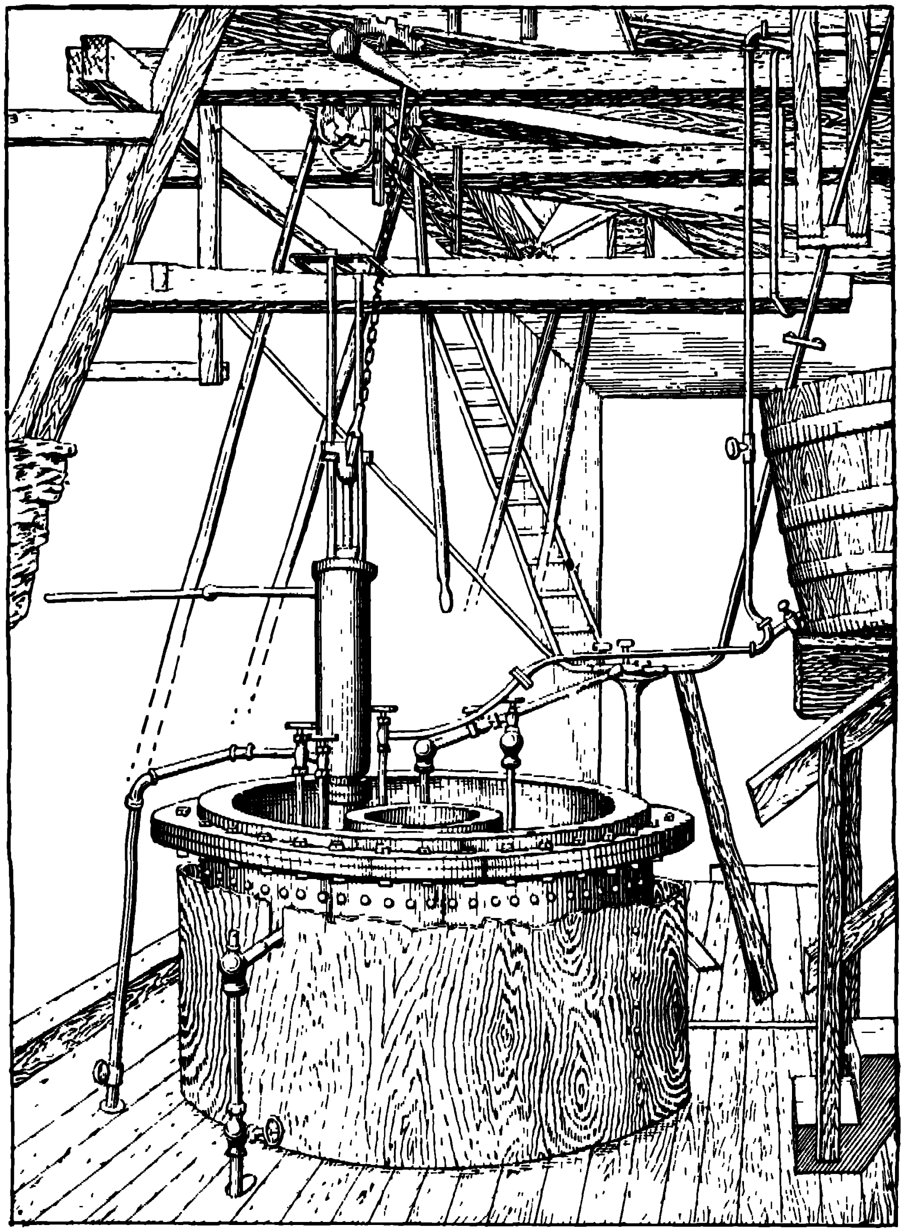

FIG. 167.—Top of Digester, Showing the Various Connections

10 hours, during which period the first charge of water is drawn off and a second charge of water, about one hour before the completion of the digestion, is introduced.

"Within the interior tank, about 5 feet from its top, is placed a perforated diaphragm, and also within said interior tank is a pump, which during the process is constantly at work, and the function of which is to preserve a constant agitation and intermingling of the contained contents, by pumping the water from the bottom to the top of the vessel and discharging it on the upper surface of the fat, in order that it (the water) may, by its greater specific gravity, shower itself through the fat."

Operation.—As already stated, the lime-saponification process is to be preferred where glycerin is to be recovered. To derive the maximum amount of glycerin, stock of good quality should be used. The utilization of the fatty acid, whether for direct pressing or distillation, determines the use of the iron or copper autoclave, for in contact with the former under high temperature and pressure discoloration of the fatty acids is effected. The stock to be used is stored in tanks, from which it may be delivered by gravity or suction to the digesters. *LL*, in Fig. 166, indicate lime-water tanks in which lime to the extent of 1 to 3 per cent of the stock used is reduced to a consistency whereby it may be discharged into the digesters *DD*; *A* is an engine which through the gears *BB* and cranks *CC* operates the pumps in the digesters *DD*; *PP* are the steam-cylinders of the agitating pump of the digesters *DD*, also shown in Fig. 167; *E*, *F*, and *G* are pipes for the admission of water, stock, and steam; *P* is a pump. The charge of fat with the regulated amount of water and lime is introduced into the digester; steam is admitted and agitation is begun. The period of saponification varies with the amount of stock and the temperature and ranges from 125 to 200 pounds steam pressure for 8 to 10 hours. The rate of decomposition is more rapid during the first few hours of the period; the rate throughout the period varies greatly.

Analyses of stock at each hour of the period made by Lewkowitsch indicate the rate of decomposition for tallow limed 3 per cent to be as follows:

TABLE XXVII.—RATE OF HYDROLYSIS OF TALLOW IN AN AUTOCLAVE (DIGESTER).

	Free Fatty Acids. Per Cent.
Sample taken after the 1st hour contained	38.5
“ “ “ “ 2d “ “	77.4
“ “ “ “ 3d “ “	83.9
“ “ “ “ 4th “ “	87.5
“ “ “ “ 5th “ “	88.6
“ “ “ “ 6th “ “	89.3
“ “ “ “ 7th “ “	93.0
“ “ “ “ 8th “ “	97.5
“ “ “ “ 9th “ “	98.1
“ “ “ “ 10th “ “	98.6

A greater or less percentage of neutral fat escapes decomposition. At the expiration of the period, of a definite length for stock of a given quality under suitable conditions of temperature, pressure and agitation, steam is turned off and the contents allowed to settle a short time, whereupon the fatty acids separate from the sweet-water, which after sufficient subsidence is blown by steam through the three-way cock into wooden tanks. The fatty acids are discharged through the same cock through a separate pipe into lead-lined wooden tanks. After the discharge of the first sweet-water from the digester, fresh water may be admitted and digestion and agitation continued for an hour. This procedure not only extends the period of decomposition under conditions more favorable to effect the last stages of decomposition, but serves to wash out the glycerin mechanically retained by the fatty acids as well. The separation of the weak sweet-water and fatty acids is effected as before.

Treatment of Sweet-water.—The products of digestion are sweet-water containing from 10 to 15 per cent of glycerin, according to the proportion of water introduced into the digester with the stock, the quality of the stock and its degree of decomposition, with more or less fatty matter, according to the thoroughness of separation, and a mixture of lime-soap and fatty acids with a small percentage of neutral fat. Sweet-water as it comes from the digester, where decomposition has been effected, is not a clear liquid, but is an emulsion of fatty acids and aqueous solution of glycerin, in which emulsion the suspended and emulsified fatty acids often amount to twice as much as the glycerin. To separate the fatty acids from the emulsion, it is generally customary to evaporate the liquid in open vats, and to collect the fatty acids from the top of the liquid by skimming as the separation is effected by the concentration; but this separation is imperfect, as it still leaves more or less fatty acids in the crude glycerin. Such a process of the evaporation of the emulsion is expensive and obnoxious, because the odor developed thereby is very disagreeable and penetrating, besides which an inferior glycerin is obtained because some of the fatty acids dissolve in the concentrated glycerin. The emulsion may be broken up by the addition of a small amount of sulphuric acid. One-tenth of 1 per cent or less by volume of 66° Bé. sulphuric acid will be sufficient. This is added to the glycerin-water while hot, which is then allowed to stand until cool enough for the fatty acids to form in a solid layer upon the top, leaving a perfectly transparent liquid containing the glycerin below. For this separation twelve hours will generally be found to be sufficient. Under some circumstances, and in warm climates, the acidulated sweet-water may be cooled by artificial means, such as by ordinary cooling-pipes within the tank, and through which cold water may be circulated. The fatty acids are then removed or the sweet-water is drawn off. To the sweet-water, which is now slightly acid, slaked lime is added in sufficient quantity to neutralize, or preferably a little more than neutralize, the acid. The thorough admixture of the lime is secured by any usual means, such as by blowing air through the liquid. The neutralized liquid is then filtered or clarified by settling and decantation. The resulting liquid will be clear and ready for evaporation in any approved form of evaporating apparatus. As it contains no mineral salts and is very free from organic impurities according to the quality of the stock its concentration is much simpler than that required for waste soap lye. For this purpose triple-effect evaporators may be economically used. The product of concentration, called "saponified crude glycerin," is of higher quality than the corresponding grade obtained from waste soap lye, as the percentage of organic and inorganic impurities being much less, a greater yield of refined glycerin may be obtained on distillation. For distillation the same apparatus and procedure as will be described for crude glycerin obtained from waste soap lye may be used.

Treatment of Fatty Acids.—The mixture of fatty acids and lime soap blown by steam from the digesters into lead lined tanks is heated by live steam with a sufficient quantity of dilute sulphuric acid to decompose the lime soap. The contents of the tank resolve themselves into three portions, viz., fatty acids, wash-water containing some free acid and gylcerin, and insoluble sulphate of lime. The fatty acids are decanted into a separate tank and are washed with fresh water to thoroughly eliminate all traces of acid. The acid wash-water of the decomposing-tank may contain sufficient glycerin to warrant its recovery, in which case it may be used to neutralize the alkalinity of waste soap lye, if a soap-factory be operated in connection. With fatty acids obtained by lime saponification from inferior stock, subsequent distillation is necessary to obtain a white and inodorous product, but when from good stock the separation of the fatty acids into their solid and liquid constituents may proceed at once. The fatty acids are composed of a mixture of stearic, palmitic (which are solid at ordinary temperature), and oleic (liquid) acids, with a solidifying-point varying from 39° to 44° C., according to the relative proportions of solid and liquid fatty acids, the solidifying-point of the fatty acids being invariably somewhat higher than the melting-point of the corresponding glycerides.

Graining or Seeding.—The mechanical separation of oleic from stearic and palmitic acids, in the case of tallow, is based upon the different melting-points of the fatty acids, and is effected by allowing the melted mixed fatty acids to cool slowly in shallow tinned or enamelled iron trays arranged one above the other in racks and filled successively from top to bottom. Each alternate tray projects over the other and permits the liquid when one tray is filled to discharge by gravity into the one beneath, the series being fed from an overhead trough directly into the top tray. The time of crystallization or seeding depends upon the temperature and varies from twelve to twenty-four hours. Crystallization should be allowed to ensue slowly so that the crystals of stearic and palmitic acids formed may be large and retain loosely the liquid oleic acid. With rapid crystallization, the crystals formed are small and closely interlocked and not permitting the freest separation of the liquid acid on pressing. The proportion of solid and liquid fatty acids is an important consideration and for the best results the solid acids should not be less than 60 per cent of the whole. Where palm-oil is saponified its fatty acids are usually mixed with those from tallow in the proportion of two parts of the former to one part of the latter. A view in the seeding- or cooling-room is shown in Fig. 168.

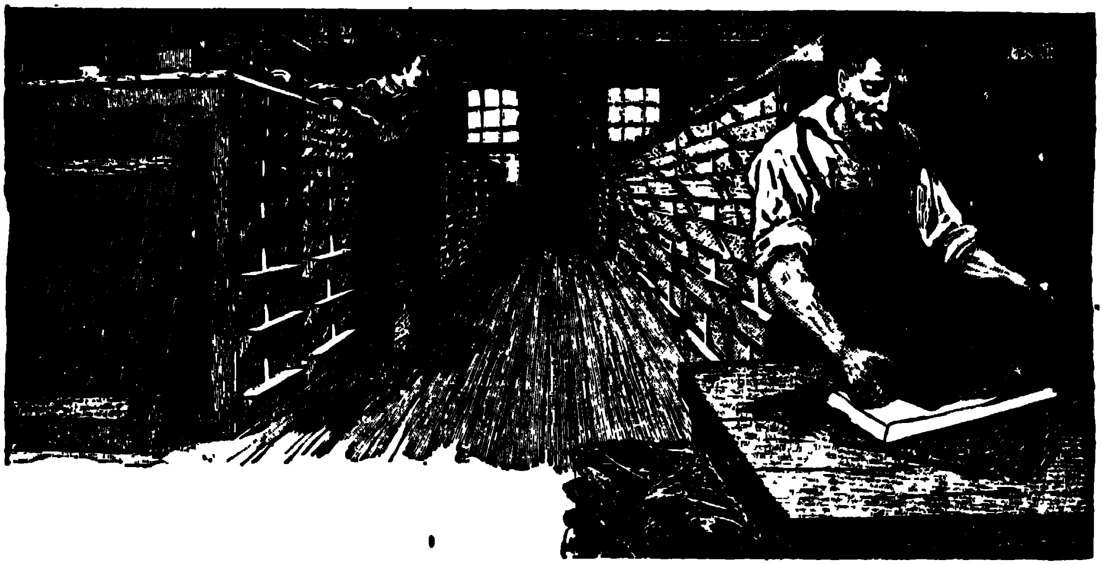

FIG. 168.—Cooling-room.

Cold-pressing.*—On cooling, the contents of the tray are turned upside down on a coarse woollen press-cloth the ends of which are folded evenly over the cake, the whole resting upon an iron plate which is placed with its burden upon a previously wrapped cake in a hydraulic press, a type of which is shown in Fig. 169. This procedure continues until the press is filled, whereupon pressure is applied very slowly, and generally in two or three stages, in the first of which the cakes "set" and permit of the uniform distribution of pressure and in the later stages the pressure is applied more strongly. A press outfit comprises either a belt or steam hydraulic pump, which transmits pressure to the hydraulic cylinder, where it is magnified according to the ratio existing between the diameter of the press-ram and that of the pump-plunger and transmitted to the material to be pressed. During the application of pressure the oleic acid flows from the cakes into a tank directly beneath, from which it is transferred from time to time to storage-tanks. Pressure is applied successively at 1000, 2000, and 3000 pounds per square inch. When the flow has ceased, the pressure is relieved, the press-ram lowered, and the cakes removed. The reduction in size depends upon the proportion of oleic acid expelled and is, as a rule,

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* For construction and operation of the hydraulic press, see "Cottonseed Products," by L. L. Lamborn.

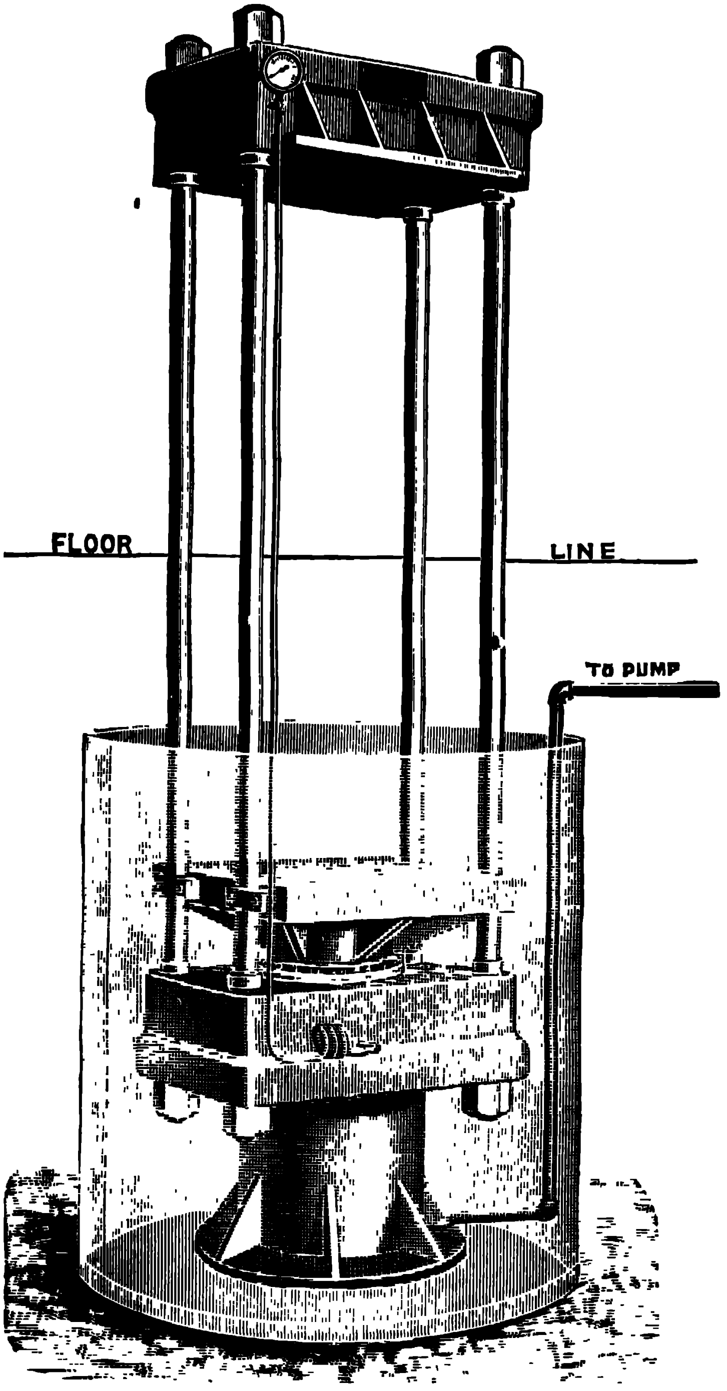

FIG. 169.—Cold-press.

about two-thirds to one-half of the original bulk. Oleic acid, to an extent of 45 per cent (this figure, however, is variable) is expelled by cold-pressing. A portion is yet retained which is expelled by hot-pressing. The yield in cold-pressing depends upon the consistency of the cake and the temperature of pressing. When pressed too warm there is a considerable discharge of stearic acid into the liquid acid. When pressed too cold, or with insufficient pressure, less oleic acid is removed and remains to be expelled in hot-pressing. The yield of liquid acid from the mixed fatty acids obtained by acid distillation is much less than from that obtained by lime saponification. Stearic acid may be recovered from the liquid acid by cooling the latter and separating the portion thus crystallized by means of a filter-press. The portion thus recovered may be either hot-pressed at once or subjected first to cold pressing, according to its consistency.

Hot-pressing.—The cakes are removed from the cold-press and placed between the hollow steam-heated plates of the hot-press shown in Fig. 170. Hot presses are horizontal, while the cold-presses may be either vertical or horizontal; the former type for cold pressing is the one generally used in the United States. The hot-press is operated in the same manner as the cold-press, i.e., by hydraulic power. A safety-valve provides against an excess of pressure above the maximum for a given temperature. Steam is admitted to the plates from a horizontal pipe above the press by means of upright pipes connected with each plate and permitting of a lateral movement of the latter by means of a sliding steam-joint. The hot-press is opened and the cold-pressed cakes are inserted between the plates until the press is filled, whereupon steam is slowly admitted to the plates until they become heated to the temperature desired to effect hot-pressing for any given stock, usually, however, between 140°–150° Fahr. Pressure is then admitted to the hydraulic cylinder, which is magnified, and transmitted to the cakes between the heated plates. The usual maximum pressure in hot-pressing is 3000 pounds per square inch. The results obtained in hot-pressing are determined by the temperature and pressure. To expel the last portion of oleic acid, as a rule about 10 per cent of the total acids, more or less stearic acid must be expelled with it. The liquid as expressed flows into a tank beneath the press, from which it is

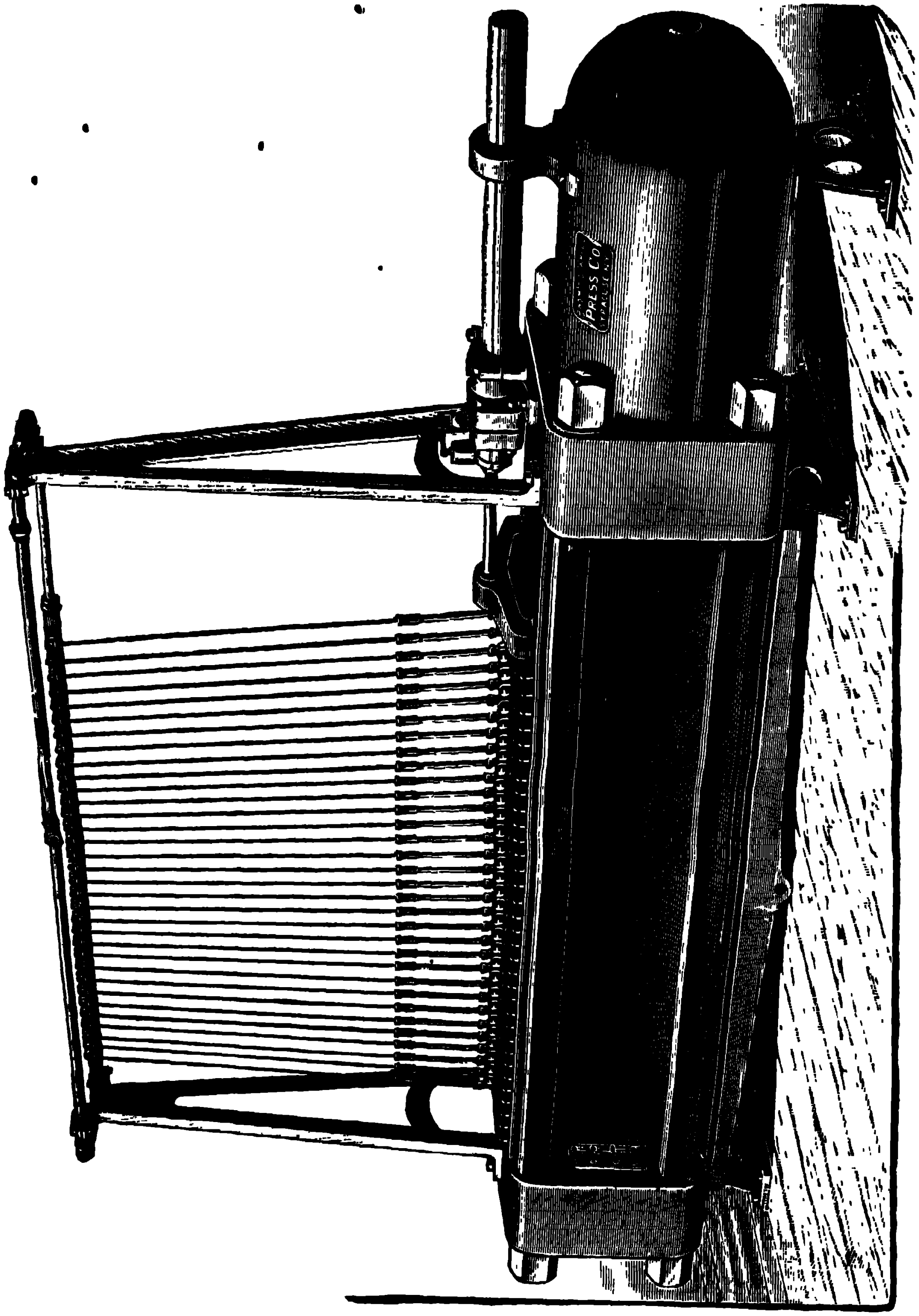

FIG. 170.—Hot-press.

removed and may be either cold-pressed separately or added to the liquid fatty acids before seeding.

The rapidity of operation depends upon the number of plates. With a thirty-plate press, about one pressing may be made in an hour, although it is subject to much variation according to conditions. After the cessation of the flow for the temperature and pressure allowed, the pressure is relieved and the plates swung back one by one to permit of the removal of the cakes, which when the press is emptied are stripped and the cloths returned for use at the cold-press. In hot-pressing the plates may be covered with horse-hair matting or the cold-pressed cakes inserted in horse-hair bags. On stripping the cakes are carefully examined for discoloration, which portions are trimmed off to be regrained and pressed. To insure the complete separation of oleic acid the cakes may be remelted and repressed. This may occur after cold-pressing, but more commonly after hot-pressing when such procedure is followed. The material thus treated produces a more durable candle, burning with greater brilliancy. The stearic acid free from discoloration or impurity is ready for moulding.

Utilization of Fatty Acids.—In the lime-saponification process the stock was resolved into fatty acids mixed with some lime soap and sweet-water containing glycerin. The latter was purified, concentrated, and distilled, producing the refined glycerin of commerce. The fatty acids and lime soap were treated with dilute sulphuric acid and after washing and decantation the entire fatty acids were crystallized or seeded and pressed first cold and finally hot to separate completely the liquid oleic from the solid stearic and palmitic acids. Oleic acid, commonly called red oil, finds application in the arts chiefly as a wool oil and as a soap-stock. The solid fatty acids are the raw material of the candle-moulder, by whom, either alone or in admixture with varying proportions of paraffin, beeswax, etc., they are utilized. Other uses are very limited.

Acid Saponification.—Chevreul in 1824 was the first to show that fats on treatment with sulphuric acid are decomposed into fatty acids and glycerin. The process is applied only to stock of inferior quality such as dark-colored fats and oils recovered from garbage, hides, and other industrial sources, the recovered product from its nature being valueless as a source of glycerin and wholly unfitted for treatment by lime saponification. The product of acid saponification is dark-colored and odorous and is purified by distillation, the two processes thus being associated. Saponification with acid and the distillation of the resulting product are described in the following sections.

Use of Process.—In acid saponification followed by distillation the glycerin is practically destroyed. As this process is used only for inferior stock and is essentially a purifying process, the loss of glycerin is inconsiderable. Considering the inferiority of the raw material and its low yield of glycerin it would not be economical to treat it for glycerin. The loss of fatty acids is considerable, varying with their quality and the skill of operation. The liquid hydrocarbons contaminating red oil and the solid candle pitch represent in part the decomposition of fatty acids and in part the concentration of organic impurities. A greater yield of solid fatty acids is obtained by this process than by lime saponification. By the latter process tallow will yield about 45–47 per cent of solid fatty acids; by the former 55-63 per cent, but with slightly lower solidifying-point. With respect to oleic acid the comparative yield is about 47 48 per cent by the lime-saponification process and about 30 per cent by the acid-distillation process. The increased yield of 15–17 per cent of solid fatty acids by the latter process may be explained by the action of concentrated sulphuric acid upon the unsaturated oleic acid, whereby it is converted chiefly into stearolactone and certain isomeric forms of oleic acid having a higher melting point than the unconverted oleic acid.

Lime saponification is adapted for good stock and where it is desired to recover glycerin, for by this process the entire amount (10 per cent) may be obtained. Acid saponification and distillation are essentially a purifying process and adapted only for inferior stock. Where inferior stock is of value as a source of glycerin, the glycerin may be recovered by lime saponification and the fatty acids thus obtained distilled, permitting the increased yield of fatty acids as well as the recovery of glycerin.

Acidification.—The dark-colored fat is first melted in an iron tank provided with a closed steam-coil, whereby impurities are allowed to subside and are removed and any retained water expelled by evaporation. The temperature may range from 200° to 300° Fahr. The fat thus purified is now ready for acidification. The high temperature and strong acid preclude the use of an iron vessel. Where acidification is effected at a comparatively low temperature, a lead-lined vessel may be used, but where a high temperature is employed the vessel is preferably of copper and closed and provided with an aspirator for the removal of vapors. In the conventional diagram of the apparatus employed in the acid-distillation process shown in Fig. 171, the acidifier *O* is represented as a closed copper vessel surmounted by the lead-lined tank *E*, from which the requisite quantity of sulphuric acid (3 to 6 per cent, of specific gravity 1.82–1.84) flows by gravity into the acidifier. The contents of the acidifier are heated by a closed copper coil through which steam superheated in the furnace *F* is made to circulate. Sulphurous vapors incident to the reaction between the fat and acid are aspirated from the acidifier through the pipe *M* by means of a water-jet in the vertical pipe discharging into the chamber *d*. The acidifier receives its charge of stock from the storage-tank by means of the pump *C* through the pipe *c*. Heating of the mixed fat and acid continues from two to five hours, during which the mixture assumes a very dark color; the sulphuric acid undergoes reduction with the evolution of sulphurous fumes and the separation of tarry matter ensues from the decomposition of a portion of the fatty acids. The mixed fatty acids are converted into sulphonated acids. On completion of the reaction the mixture is allowed to stand to deposit tarry matter, whereupon the supernatant fatty acids are discharged into a lead lined reservoir represented in Fig. 171 by the series *G*

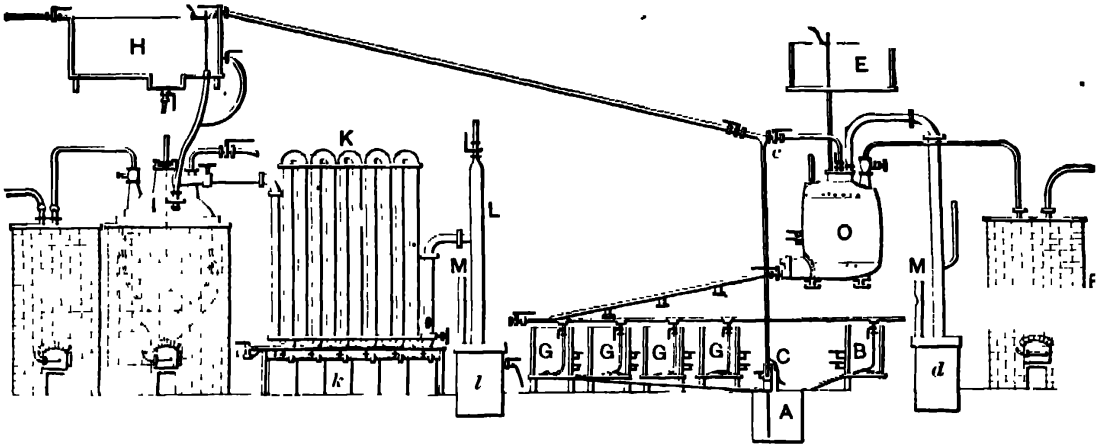

FIG. 171.—Apparatus of the Acidification Process.

of lead-lined tanks provided with copper open steam-coils. The acid foots remaining in the acidifier consists of tarry matter and sulphuric acid mixed with fatty acids. The whole is discharged from the acidifier and the latter separated and added to the main body of fatty acids. The sulpho or sulphonated fatty acids are boiled with water acidulated with sulphuric acid by means of live steam for the purpose of decomposing the unstable sulpho-compound, whereupon after complete decomposition the acid water is settled out and removed and the liberated fatty acids are transferred to the storage-tank *H*, above the still *I*, from the reservoir *A* by means of the pump *C*. The acid-washing process subsequent to acid saponification serves not only to decompose the sulpho-fatty acids but to remove tarry matter carried in suspension from the acidifier; a second boiling with water and a little sulphuric acid may be required to effect the degree of decomposition and purification desired.

Owing to the powerful dehydrating effect of concentrated sulphuric acid great care must be used in regulating its amount and density and the temperature to which the stock is heated. Sulphuric acid serves primarily as an accelerator of the hydrolysis; in its subsequent combination with the liberated fatty acid to form the unstable sulphonated compound it plays a rôle analogous to caustic alkali in soap boiling, though in the latter case the compound formed is stable. With an insufficient amount of acid or with an acid of too low a density hydrolysis is incomplete, the same decreasing as the density of the acid is reduced until it becomes nil with acid containing less than 60 per cent of H_2SO_4 (49°–50° Bé.). The higher the temperature to which the stock is heated, and the greater the proportion of acid used, the lower will be the yield of fatty acids, and of glycerin if this warrant recovery. Abnormal conditions in these respects are indicated by the copious evolution of sulphurous fumes and by the excessive destruction of glyceride and non-glyceride matter by carbonization. It should be endeavored to effect the most intimate admixture of the stock and acid of proper density and amount in the shortest time.

Construction of Still.—The distillation of fatty acids by means of free steam dates from 1842–43, at which time (*vide* page 443)

Wm. C. Jones and George F. Wilson patented a process which superseded a previous invention (1840) for their distillation *in vacuo*. This process originating with the precursor of the Price's Patent Candle Co., Ltd., remains, with practically little, if any,

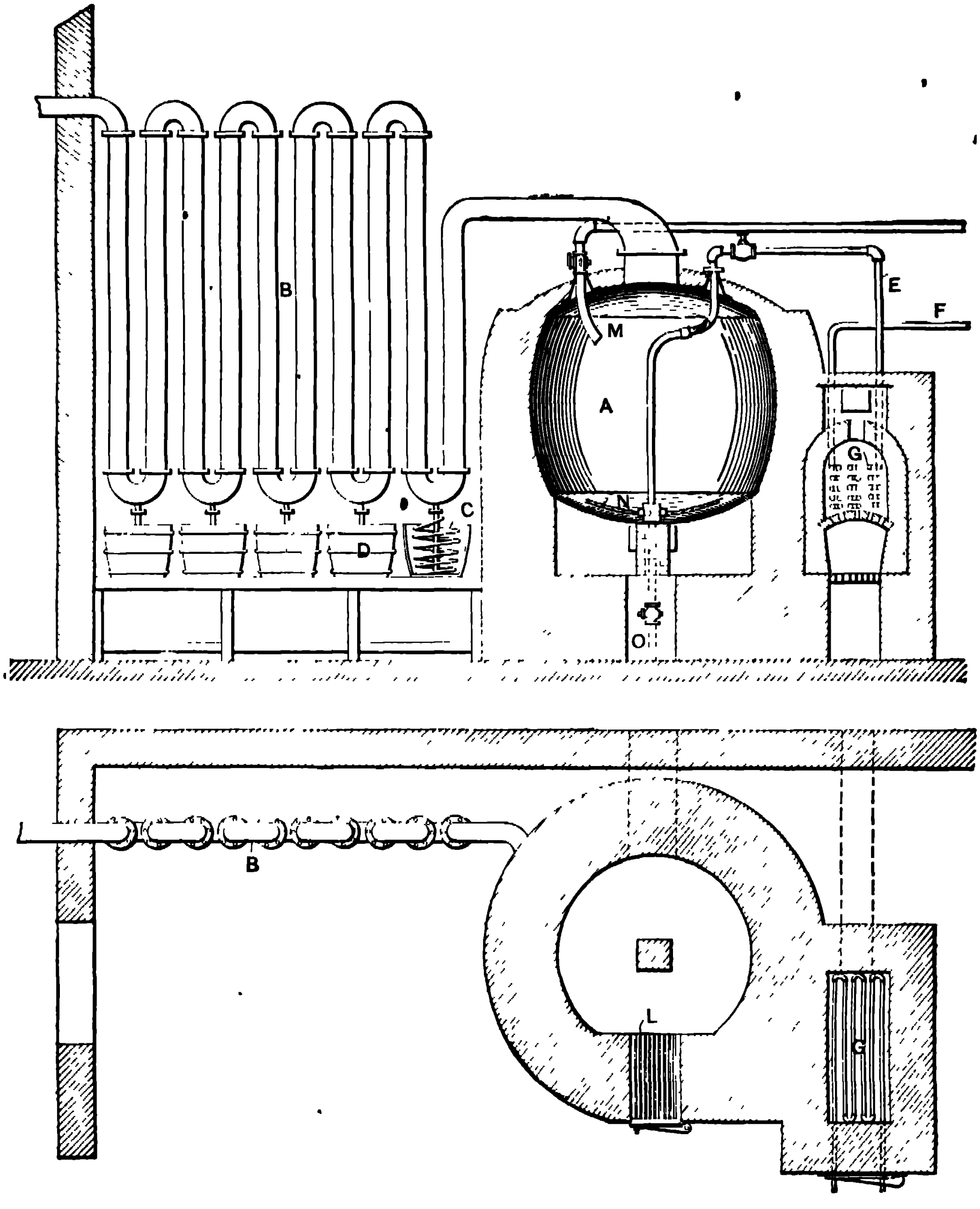

FIG. 172.—Type of Still for Distillation of Fatty Acids—Plan and Elevation.

essential modification, the recognized method for the distillation of fatty acids. By reference to Fig. 171, showing the conventional diagram of the acid-distillation process, the essential elements of the still equipment are shown; *H* is a lead-lined tank placed at a sufficient elevation above the still, whereby the fatty acids stored therein may be allowed to run by gravity into the copper still *I* as required. In Fig. 172 is shown in plan and elevation a still for fatty acids in actual use. *A* is a copper still; *B* a condenser; *C* is a copper coil contained in the wooden tubs *D*; *E* a steam-pipe from the superheater *G*; *F* a steam-pipe from the boiler to the superheater *G*; *M* is a copper charging-pipe for introducing fatty acids into the still *A*; *N* is a copper open steam-coil for injecting superheated steam in the mass of fatty acids contained in the still *A*, which is heated externally by fire upon the grate *L*; *O* is a blow-out pipe for the residuum, or pitch, remaining after distillation. An auxiliary condenser, not shown, is located out of doors for condensing the lighter portions of the redistilled tar. The copper coils shown at *C* are surrounded by cooling-water and are a continuation of the pair of upright condensers, with which they are respectively connected. The distillate is discharged from the copper coils into separate tanks, according to its quality. The condensers, as a rule, are of copper, the last one connecting with a suitable aspirator (see *L*, Fig. 171), whereby vapors are condensed and carried away by water discharged into tank *l*. Vapor yet uncondensed is conveyed through *M* and suitably disposed of. The condensers may be water-jacketed entire, or only the last one or two, to effect condensation of the more volatile portions of the distillate; as a rule, however, air cooling suffices, for which purposes the condensers may sometimes be placed outside of the building.

Distillation of Fatty Acids.—Fatty acids for distillation may be either those obtained by lime saponification, but unsuited owing to their dark color, for immediate pressing, or those obtained, as previously described, by acid saponification and intended for distillation. The first charge of fatty acids is introduced into the still and heated by direct fire. Superheated steam in closed coils may be used to avoid danger of charring incident to direct heat, but the former method is more common. When the contents are heated to a temperature approaching that of distillation, viz., about 500° Fahr., superheated steam at about the same temperature is injected through the copper open steam-coil. The temperature of distillation varies, but to secure continuous distillation and a distillate of good color, it should not depart from 500° to 520° Fahr. The quantity of fatty acids distilled during a single "run" of the still varies with its size; with a still of ordinary size 16,000 to 18,000 pounds may be treated before shutting down. During distillation the volume in the still is kept constant by the continuous introduction of fresh material from the storage-tank above as rapidly as it distils over. The distillate, as a rule, is separated into three fractions. The first fraction, comprising about 80 per cent of the entire distillate, is of white color and may be pressed at once. During distillation more or less decomposition of fatty matter occurs with the formation of bodies of simpler composition, viz., hydrocarbons. These for a part have a higher boiling-point than the associated fatty acids and remain in increasing amount in the still. Their presence in the distillate is shown by a green color, which, when it becomes decided, indicates that the distillate should be separated into the second fraction. This fraction is returned to the storage tanks for fatty acids and is redistilled with the next run. As distillation proceeds the color of the second fraction becomes darker and more viscid, at which stage distillation is stopped whereupon the contents of the still are transferred to a smaller iron still permitting the application of higher heat. The distillate from this still is collected in one portion, and, as a rule, is returned to the storage-tank for redistillation. It is highly charged with unsaponifiable matter, which, when the distillate is disposed of in this manner, remains to a large degree to contaminate the red oil. After separation of three portions of the distillate, as above described, there remains in the still a black residuum, viscous when hot, but solidifying on cooling to a hard, brittle mass, known as candle-pitch, or tar,

Products of Distillation.—To determine quantitatively the changes wrought by the high temperature of distillation, viz., about 500° Fahr., in the composition of the fatty acids, a run* of 16,075 pounds was traced through, and the chemical composition before and after noted, as well as the proportion of the different grades of oil above named. These figures may be taken as typical, the variation from averages being very small.

W. E. Garrigues in a paper before the American Chemical Society

The mixed fatty acids had a melting-point of 43.2° C., and contained:

	Before Distillation, Per Cent.	After Distillation, Per Cent.
Solid acids.	49.52	50.47
Oleic acid*	42.86	33.05
Hydrocarbons (liquid) †	6.94	13.14
Glycerin ‡	1.37	
Tar.	.....	2.00
Loss	.....	1.34
	100.69	100.00

The percentage yield of each distillate and its constitution were as follows:

	Per Cent.			Per Cent.
First distillate	80.31	Containing:	Hydrocarbons	6.29
M.-pt. 44.8° Fahr.			Fatty Acids	93.71
Second distillate	12.13	Containing:	Hydrocarbons	35.90
			Fatty Acids	64.10
Third distillate	4.22	Containing:	Hydrocarbons	98.74
			Fatty Acids	1.26
Tar	2.00			
Loss	1.34			
Total	100.00			

Making corrections for the hydrocarbons present before distillation, and the glycerin, which forms a large part of the 1.34 per cent loss, we have as a result of distilling 100 parts of pure fatty acids:

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* All the percentages of oleic acid were obtained by calculation from the iodine absorption figure.

† Liquid hydrocarbons present in the fatty acids before distillation arise from the addition of the second and third fractions of the distillate to the fatty acids for redistillation.

‡ One-half the glycerin found is subtracted, together with oleic acid and hydrocarbons, from 100 to determine the solid acids, since tallow glycerides yield approximately 95 per cent fatty acids and 10 per cent glycerin.

	Before	After.
Solid acids	53.61	54.63
Oleic acid	46.39	35.77
Hydrocarbons (liquid)	.....	6.71
Tar	.....	2.15
Loss	.....	0.74
	100.00	100.00

It seems reasonably assured from these figures that the stearic and palmitic acids distil without change, while the oleic partly splits up into liquid hydrocarbons, tar, and a saturated or non-iodine-consuming fatty acid.

From the foregoing facts it appears to the writer that the present process of manufacture might be advantageously altered by taking the fatty acids directly from the autoclave to the presses—as is done with the light-colored stock—and obtaining a press-cake melting at 55°–56°, which latter is then subjected to distillation. By these means the candle-stock would not deteriorate in the still as it now does, and the formation of valueless—and even for most purposes harmful—hydrocarbons would be avoided, giving the red oils a greater worth.

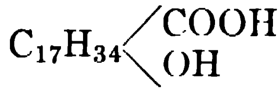
Practical candle-makers find that distilled stock invariably yields a press-cake of lower melting-point than that from the autoclave. This is now explained by the formation of the saturated fatty acid from oleic acid in the still. This body is sufficiently solid to resist being pressed out with the oleic acid, yet soft enough to lower the melting-point of its associates—stearic and palmitic. In passing, it may be pointed out, as shown by several writers, that the lowering of the melting-point of a mixture of two fatty acids by the addition of a third does not necessitate the latter having a melting-point below that of the two former.

Apart from the evidence already given, its presence is indicated by its low molecular weight, being—probably considerably—under 268, while the mean of stearic and palmitic as occurring in tallows is 271–272.

Utilization of Products.—On the distillation of mixed fatty acids the first portion of the distillate, constituting about four-fifths of the whole, is ready for pressing at once. As already described for fatty acids obtained by lime saponification, they are seeded, cold-pressed and hot-pressed, the solid fatty acids being available for candle-stock, while the liquid oleic acid is used in soap manufacture and as a wool-oil. From its origin and mode of preparation red oil obtained by acid saponification and distillation commands a lower price than that obtained by lime saponification. Red oil obtained by the latter process is called "saponified olein," while that obtained by the former is called "distilled olein." The second portion of the distillate, constituting about 12 per cent of the whole, is returned to the fatty-acid storage-tank for redistillation; likewise the third portion, although this procedure may be regarded as reprehensible. Candle-pitch, or tar, also called "stearin-pitch," constitutes about 2 per cent of the whole. It is much used for making insulating compositions for electrical purposes, for preparing water-proof paper, in the preparation of lubricating greases, and for paints for various purposes. It is a black homogeneous pitchy looking mass; about 21 per cent is soluble in alcohol, the solution being faintly acid and fluorescent. It contains about 9 to 10 per cent of saponifiable matter. On distilling it yields about 80.5 per cent of volatile oils, while 19.5 per cent of solid carbonaceous matter remains behind. The earlier portions of the distillate contain acrolein, the others varying in specific gravity from 0.7861 to 0.8877, are of a yellowish-brown color and strongly fluorescent. The heavier fractions, which are the most abundant of the number, are about three times more viscid than water and deposit hydrocarbons of a similar nature to vaseline, a feature of some interest but not at all unexpected, considering the relationship which exists between the paraffin and olefin hydrocarbons and the fatty acids. It may be mentioned that Engler and Hofer consider that petroleum has been derived by the natural decomposition of animal fats.

Manufacture of Hydroxystearic Acid.—Oleic acid in a pure state, which is difficult to obtain, is a colorless, odorless liquid solidifying at 4° C. (39.2° Fahr.) and melting at 14° C. (57.2° Fahr.); commercially its composition is determined by its origin. It is the representative acid of the oleic series of unsaturated compounds which are characterized by the property of absorbing under suitable conditions two atoms of chlorine, bromine, or iodine, or one molecule of their corresponding hydrogen acid, becoming converted thereby into derivatives of acids of the acetic (saturated) series, of which, from the standpoint of the soap- and candle-maker, stearic acid is the most important representative. It is possible by well-known chemical reactions to transform oleic acid into stearic acid; also into an isomeric body possessing the property of crystallization. On treatment with cold concentrated sulphuric acid, oleic acid forms an addition compound, sulphostearic acid, which on boiling with water separates sulphuric acid and forms hydroxystearic acid and a small amount of stearolactone. Zinc chloride acts similarly on oleic acid. Since oleic acid forms such a large proportion of the products obtained in the decomposition of fatty bodies and is unfit in its untreated state for use as candle-stock much attention has been devoted to the practical application of the numerous chemical reactions which are capable of producing from the liquid commercial acid either an isomeric, addition, or substitution product of a higher melting-point than the original body. The stability required of such a product by the conditions of its use and the great cheapness with which the process must necessarily be worked limit the use of processes that experimentally are very satisfactory.

The following process, which is valuable as indicating the limitations of available processes, is the latest modification of the most practicable process applicable for increasing the use of oleic acid as candle stock.*

It is desired to produce hydroxystearic acid,  $C_{17}H_{34}\langle{}^{COOH}_{OH}$, from oleic acid, $C_{17}H_{33}COOH$. In carrying out this process oleic acid is dissolved in petroleum distillate, which is unacted upon by sulphuric acid, in the proportion of one part by volume of acid to two parts by volume of petroleum distillate of 60° Bé. The purpose of solution is to increase the intimacy of contact of the reacting bodies and thereby to promote the thoroughness of the reaction. Polymerization may be effected by the use of zinc chloride or concentrated sulphuric acid. The latter agent preferably is used in the proportion of one part by weight of sulphuric acid to three parts by weight of oleic acid. The reaction is indicated by the following equations:

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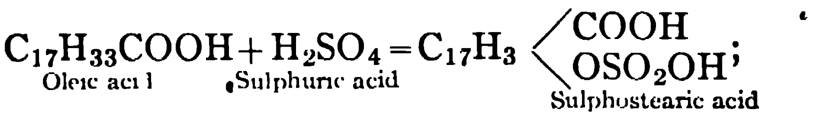
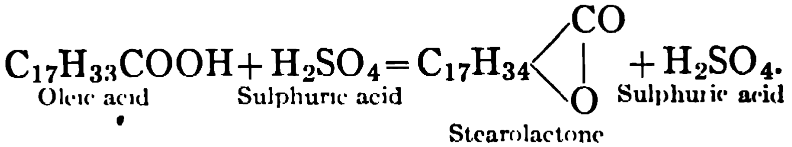
* Letters patent No 772,129. W. M. Burton, patentee; Standard Oil Co., assignee.

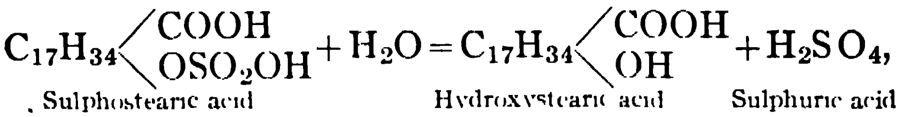


$$\underset{\text{Oleic acid}}{C_{17}H_{33}COOH} + \underset{\text{Sulphuric acid}}{H_2SO_4} = \underset{\text{Sulphostearic acid}}{C_{17}H_{3}\begin{cases}COOH \\ OSO_2OH\end{cases}};$$



$$\underset{\text{Oleic acid}}{C_{17}H_{33}COOH} + \underset{\text{Sulphuric acid}}{H_2SO_4} = \underset{\text{Stearolactone}}{C_{17}H_{34}\begin{cases}CO \\ \mid \\ O\end{cases}} + \underset{\text{Sulphuric acid}}{H_2SO_4}.$$

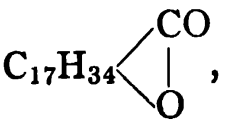
The above reactions should be effected at a temperature not exceeding 40° Fahr. The product is then subjected, under suitable conditions, to the action of live steam, whereby the sulphostearic acid is decomposed in accordance with the following equation:



$$\underset{\text{Sulphostearic acid}}{C_{17}H_{34}\begin{cases}COOH \\ OSO_2OH\end{cases}} + H_2O = \underset{\text{Hydroxystearic acid}}{C_{17}H_{34}\begin{cases}COOH \\ OH\end{cases}} + \underset{\text{Sulphuric acid}}{H_2SO_4},$$

thus producing hydroxystearic acid and regenerating sulphuric acid. The sulphuric acid is separated from the solution containing the yield of hydroxystearic acid and the associated bodies by allowing the steam-heated mass to stand sufficiently to allow a separation by gravity, whereby the mineral acid sinks to the bottom of the treatment-tank, whence it may be drawn off and suitably concentrated for repeated use. The remaining mass is then extracted under suitable conditions with an equal amount of hot petroleum naphtha, whereby the pure hydroxystearic acid is dissolved. This extract is cooled to about 40° Fahr., whereby the hydroxystearic acid separates in a white crystalline form, leaving behind the other compounds in solution. The crystalline mass after suitable filtration and drying is ready for use.

The solvent used may be recovered by distillation and used again. By the procedure thus described it is claimed that about 50 per cent of the oleic acid treated is converted into hydroxystearic acid without material loss of the reagent and solvent used. The remainder of the oleic acid, constituting about 50 per cent thereof, has been converted by the action of the reagent into stearolactone,



$C_{17}H_{34}\begin{cases}CO \\ \mid \\ O\end{cases}$, the remainder being oleic acid unacted upon. This may be recovered by suitable distillation and subjected again to the treatment described for the production of hydroxystearic acid. By thus subjecting the original charges of oleic acid to the treatment described, reconverting the residual presumed stearolactone into oleic acid and subjecting the latter to the same treatment, there is claimed to be recovered from 100 gallons of raw oleic acid from 85–90 per cent of hydroxystearic acid as compared with 25–30 per cent, which is the highest yield usually obtained by other methods. Hydroxystearic acid, of which two forms exist, viz., α- and β-, is produced by the general procedure, one form of which is described above. The relative proportions of these two isomeric forms depend upon the temperature prevailing during the reaction; the lower the temperature the greater the yield of α-hydroxystearic acid. The β-hydroxystearic acid melts at 81°–85° C. (178°–185° Fahr., and solidifies at 68°–65° C. (154.4°–149° Fahr.); α-hydroxystearic) according to different observers, melts at 77°–85° C. (170.6°–185° Fahr.). The original oleic acid melts at 4° C. (39.2° Fahr.) and solidifies at 14° C. (57.2° Fahr.).

Twitchell Process.—The Twitchell process for the decomposition of fats and oils into fatty acids and glycerin is based upon the discovery that when neutral fat to which has been added a small quantity of a sulphobenzene fatty acid is boiled with one-half its bulk of water, it undergoes decomposition into fatty acids and glycerin, which on standing separate into layers in a high state of purity. The great simplicity of this process, the ease and cheapness with which it can be carried out, and the high purity of its products make it a formidable competitor of the historical processes which have been hereinbefore described. The decomposition of the neutral glyceride is the initial problem common to both the soap and candle industry. In the former the fatty acid is combined with caustic alkali to produce soap, while the glycerin is recovered by an expensive process of purification, concentration, and distillation from a foul by-product; in the latter the fatty acid is used directly, while the recovery of the glycerin is a comparativly simple process inasmuch as the menstruum in which it is contained is a much purer body than waste soap lye. If the waste lye of the soap industry can be made as pure as the sweet-water of the candle industry, or, better, if waste soap lye as a technical raw material of a valuable body can be eliminated entirely by a process wherein the glycerin is separated in an aqueous solution of great purity before soap is made, a long step forward in the industrial efficiency of soap manufacturing processes is possible.

We are concerned at this place with the study of the raw materials of candle-manufacture, the study of glycerin being reserved for later consideration. Inasmuch as the Twitchell process has become general in the candle industry its study at this place is appropriate; and to avoid repetition, the student of soap-manufacture, in which art its field of usefulness is greater, is referred here for his facts. From the standpoint of soap manufacture the following points of economic superiority over the historical process may be indicated: (1) the greater yield of glycerin obtainable from the stock; (2) the high glycerin content and great purity of the glycerin menstruum, resulting in the greater ease and less expense of recovery of the glycerin therefrom; (3) the greater purity of the concentrated product, viz., crude glycerin and the greater ease of its distillation necessary to produce the refined grades; (4) the availability of cheaper soda-ash as a saponifying agent; (5) the greater simplicity of manufacturing equipment.

Returning to a consideration of the intrinsic nature of the process our attention is directed first to the instrumentality whereby the decomposition of the glyceride is affected.

Sulphoaromatic Fatty Acids.—The chemical reagent used in effecting decomposition by the Twitchell process is a product of the action of sulphuric acid upon a mixture of oleic acid and benzene, at a temperature below 30° C., in accordance with the following equation:

$$\underset{\text{Benzene}}{C_6H_6} + \underset{\text{Oleic acid}}{C_{18}H_{34}O_2} + \underset{\text{Sulphuric acid}}{H_2SO_4} = \underset{\text{Sulphobenzene-stearic acid}}{C_6H_4(HSO_3)(C_{18}H_{35}O_2)} + \underset{\text{Water}}{H_2O}.$$

The sulpho-fatty acids which are largely used in the arts are unstable compounds, and that for this reason in the production of the same by the treatment with sulphuric acid of a fatty acid or oil, the yield or percentage of the sulpho-fatty acid is small in comparison with the reagents employed. A large percentage of the reagents are wasted and the unstable product itself is easily decomposed and separated into its component parts.

In the preparation of this compound effort is made to overcome this objection by producing a reagent which will accomplish the functions of the sulpho-fatty compound and which will be stable under all ordinary circumstances, and which can be produced simply and with a much less waste of the reagents employed.

In the practical preparation of the compound the fatty acid and the body of the aromatic series, which may be either naphthalene, benzene, phenol, etc., are mixed in the proportions of their molecular weights, whereupon concentrated sulphuric acid is added in excess, care being taken to effect the reaction at a temperature not in excess of 30° C. This new compound obtained in this way is contaminated with small quantities of, first, unacted on oleic acid and other fatty matter; second, unacted-on benzene; third, sulpho-fatty acid; fourth, benzol sulphonic acid; fifth, sulphuric acid in large quantities. To purify the compound for chemical investigation, the discoverer and inventor treats the product of the reaction with water and heats to boiling, whereby the excess of sulphuric acid is diluted and the mass separates into two layers. The lower clear aqueous layer contains the excess of sulphuric acid and whatever benzene-sulphonic acid may have been produced. This latter compound is soluble in dilute sulphuric acid, while the new sulpho-fatty aromatic compound is not. The upper layer, a viscous oil, contains the sulphobenzene-stearic acid with the remaining impurities. The lower layer is removed and the upper layer is repeatedly washed with dilute hydrochloric acid in which the sulphobenzene-stearic acid is insoluble. The sulpho-fatty acids (not combined with the benzene) originally present in the mixture will have been decomposed by the heating into sulphuric acid and fatty matter. The oily layer is then washed a number of times with petroleum ether (commercial benzine or gasoline), which dissolves the fatty matters and the benzene, leaving the compound pure, except for small quantities of water and hydrochloric acid, and these can be easily removed by drying at 100° C. This compound has the following formula:

$$C_6H_4(HSO_3)C_{18}H_{35}O_2.$$

In place of the benzene, naphthalene and phenol have been employed and compounds obtained which in physical properties are almost identical with that obtained from benzene. The sulpho-naphthalene-stearic acid has the following formula:

$$C_{10}H_6(HSO_3)C_{18}H_{35}O_2.$$

A general formula for this entire series of new compounds would be $R(HSO_3)C_{18}H_{35}O_2$, in which R represents the aromatic radical.

For commercial purposes it is not necessary to purify the new compound as described, one washing with water being generally sufficient.

These compounds possess the characteristics of sulphonic acids. On fusing with caustic potash, potassium sulphite is obtained. They are dibasic acids. In physical properties these compounds resemble much more the sulpho-fatty acids than the aromatic sulphonic acids. They are amorphous compounds, soluble in water, but insoluble in solutions of acids or salts. They differ from the simple sulpho-fatty acids in being exceedingly stable, not decomposed by boiling with water or acid solution. This not only renders them more valuable as commercial articles, but extremely useful for laboratory purposes.

One of the chief uses of this newly discovered reagent lies in the decomposition of fats and oils into fatty acids and glycerin. There will be a series of these compounds corresponding to the members of the oleic acid series and aromatic series, which are mixed together and treated with the sulphuric acid. These compounds have uniform physical characteristics, varying slightly only in stability, according to the particular aromatic body employed, the chemical formulæ varying, of course, only as the aromatic radical varies with the particular member of the aromatic series used.

Outline of the Twitchell Process.—In general the process consists in simply boiling the fat in closely covered wooden tanks of appropriate size with water and a small percentage of the special chemical or saponifier which effects the decomposition of the glyceride. The boiling is done with open steam, and at the end of one or two boils of varying duration (according to the kind of stock in hand and the purpose for which the resultant fatty acid is to be used) the glycerin is found to be present in the water which settles to the bottom, and the fatty acids are ready to be used at once either for candle-manufacture or the production of soap. The glycerin-water after treatment with a small percentage of common lime is ready to be evaporated in any kind of evaporator, the simplest form being an open tank with closed steam-coil.

To effect complete decomposition of the glyceride and to prepare the resulting products in the best condition for subsequent utilization, certain precautions are necessary. In order that the smallest amount of the saponifying agent may be used, the fat must be freed from all dirt, lime, bone, tissue, or other impurities natural to the various fats.

This is accomplished by boiling the fat with weak sulphuric acid in a wood or lead-lined tank and then drawing off the clean, well-settled fat into another tank.

The tank into which the purified fat is run or pumped cannot be of iron, but preferably must be of wood and fitted with a simple brass, copper, or lead coil for open steam only, and have a loose cover fitting snugly but not necessarily perfectly tight.

To the fat in this second tank is added about one-third of its weight of distilled water and ½ to 1 per cent of its weight of the saponifying agent.

This mixture of fat, water, and chemical is now boiled with open steam for twelve to twenty-four hours, the time varying with the percentage of the saponifying agent used. At the end of this time nearly all the glycerin will have left the fat and will be found in the water. The mixture is then allowed to settle. The glycerin and water settle to the bottom and the fatty acids collect at the top. During the boiling no air must be allowed to come in contact with the fat through having the cover on too loosely. This precaution is to preserve the color of the fatty acids which are now ready for use either as candle- or soap-stock.

The aqueous solution of glycerin at the bottom of the tank should amount to about 60 per cent of the volume of fat and is run or pumped to a treating-tank, where it is neutralized with a little lime (about ¼ per cent) and is then boiled down in the usual evaporating apparatus, the simplest form of which is an open tank with closed steam-coil. The crude glycerin thus obtained is light-colored, nearly free from odor, and contains 85 to 90 per cent of actual glycerin.

Application of the Process on an Experimental Scale. Preliminary Acid Boil.—Place in cask *A*, Fig. 173, the fat to be treated and bring to a boil with the open steam-coil. While boiling add from 1 to 2 per cent, say 1½ per cent, of 60° Bé. sulphuric acid and boil from one to two hours. Turn off the steam and let stand a few hours or overnight for the acid-water to settle out. The waste acid-water should show a strength of 8° Bé., or over, on all stocks except cottonseed-oil and linseed-oil, in which case it should show at least 15° Bé., and preferably 20° or 22° Bé. Care must be taken that the acid used is not 66° Bé., as this strength of acid would slightly discolor the stock, while 60° Bé. acid added to a fat boiling on open steam will not harm it. (60° Bé. sulphuric acid contains about 77 per cent H_2SO_4; 20° Bé contains 21 per cent; and 8° Bé. acid 8 per cent.)

It is of the utmost importance that the waste acid drawn off be not weaker than above stated, as stock will not work well in the next part of the process if the acid-water is drawn off weaker. As a general thing there is no difficulty in getting this waste acid of sufficient strength by carefully following the method of procedure above outlined, but in case the fat is cold or the steam wet the waste acid may be withdrawn too weak, especially in the case of cottonseed-oil, when it should be 15° Bé. or over. In such cases the waste acid can be gotten of the proper strength without adding any more acid than above mentioned by heating the fat to boiling with the open steam first; turn off the steam, let it stand a half hour or so, draw off the condensed water, and again bring to boil with open steam, and when boiling add the acid and boil for from one to two hours The fat being already hot when the steam is turned on the second time, there is but little condensation; and by this manner of making the acid wash it is possible, under the most unfavorable conditions, to draw off waste acid of the above-mentioned strengths without using more than the above-mentioned percentage of acid.

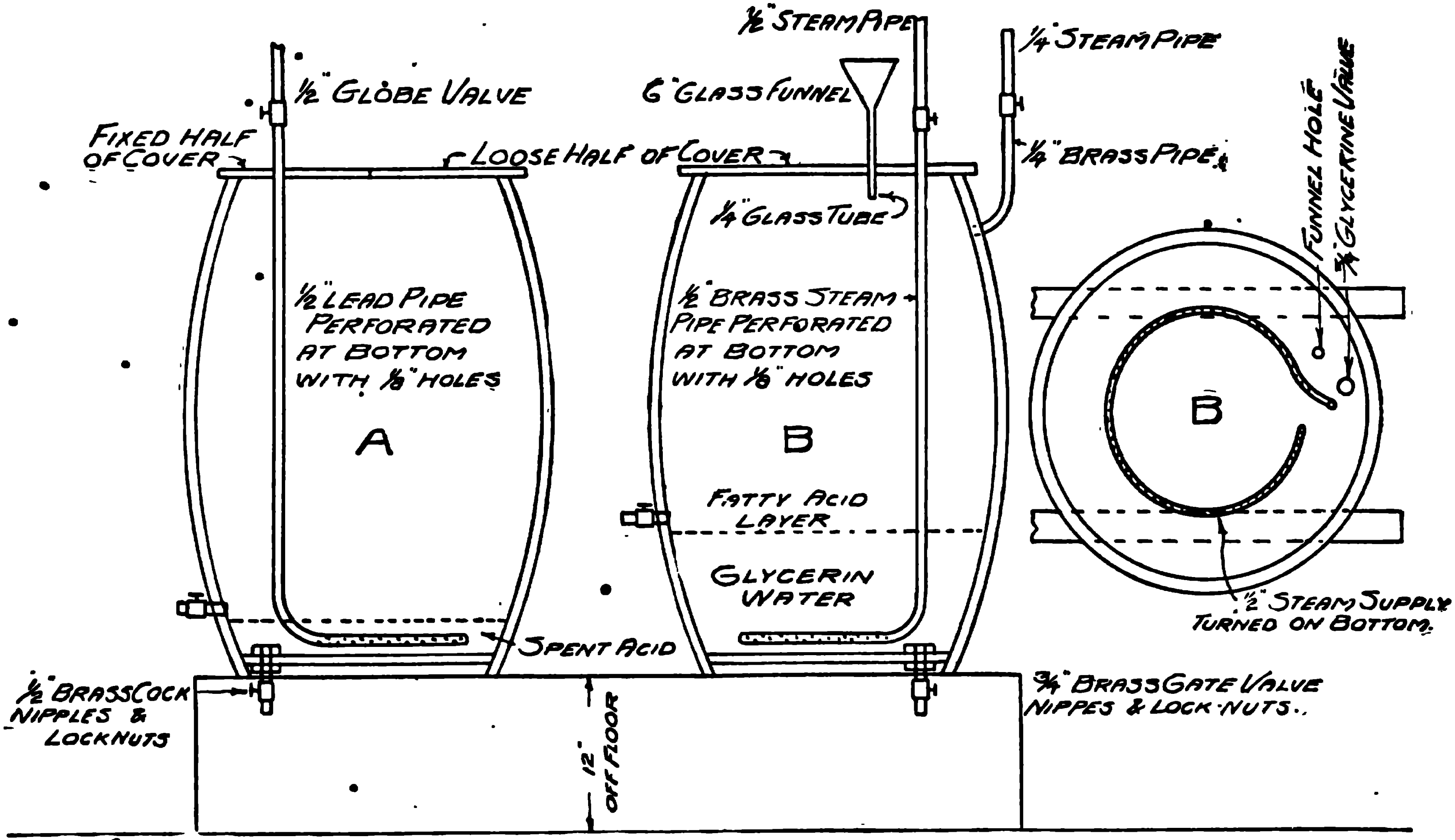

½" Globe Valve
Fixed Half of Cover
Loose Half of Cover
½" Steam Pipe
6" Glass Funnel
¼" Steam Pipe
¼" Brass Pipe
¼" Glass Tube
½" Lead Pipe Perforated at Bottom with ⅛" Holes
A
½" Brass Steam Pipe Perforated at Bottom with ⅛" Holes
B
Fatty Acid Layer
Glycerin Water
Spent Acid
½" Brass Cock Nipples & Lock Nuts
12" off Floor
¾" Brass Gate Valve Nipples & Lock-Nuts
Funnel Hole
¾" Glycerine Valve
B
½" Steam Supply Turned on Bottom

FIG. 173.—Experimental apparatus. Twitchell process. *A*, tank to boil fat on acid. (300 lbs. fat can be boiled in a 50-gal. oil-barrel; 1000 lbs. fat can be boiled in a cocoanut-oil hogshead.)

B, tank to extract glycerin. (150 lbs. fat can be extracted in a 50-gal. barrel; 500 lbs. fat can be extracted in a cocoanut-hogshead.) Cover on each tank made in halves, one tight, the other loose. Steam-supply same in each, but one pipe lead, the other brass.

The Saponification.—Five hundred pounds of the washed fat are transferred from *A* to *B* (Fig. 173), and 100 pounds of distilled water (condensed water from steam-coils) added. The steam is turned on and the contents of the tub brought to a boil. The boiling should be vigorous enough to keep the contents of cask thoroughly mixed and to cause the steam to come off freely from the surface of the liquid. After the contents of the cask have been brought to a boil, add through a glass or graniteware funnel inserted through a hole in the lid the saponifying agent weighed out in a wooden bucket and wash it all in with a little distilled water. The contents of the cask are now boiled from twelve to twenty-four hours continuously, at the end of which time most of the glycerin will have been extracted from the fat, which should show from 85 to 90 per cent free fatty acids. The steam is now turned on at the pipe, going into the side of the cask near the top, and then turned off from the coil and the glycerin-water allowed to settle out from the fat. The glycerin-water will separate nicely in a half hour on a small scale and in an hour or two on a factory scale. When a rather large amount of the saponifying agent has been used a permanent emulsion sometimes forms at this stage. A little sulphuric-acid solution (0.1 to 0.2 per cent) will break this emulsion and cause the mixture to settle. During this period of settling steam is allowed to enter through the pipe outside of the cask near the top to prevent air from coming in contact with the fat, as the contact of the air at this stage would discolor the fatty acids. The amount of glycerin-water present should be between 50–60 per cent of the fat. If more than 100 per cent is present it will be necessary to use less water at the start, and if less than 50 per cent is present it will be necessary to use more water. The first two or three runs will show for each particular plant just how much water must be added at the start, so that with the condensed water produced by the steam the total amount of water present at the end of the boil will be between 50–60 per cent of the stock. The glycerin-water should not test over 5° Bé. (about 15 per cent of glycerin). After the glycerin-water has been drawn off about 50 pounds of fresh water are added and the steam turned on the coil and contents of cask brought to a boil. During all this time of settling, drawing off, and adding water, the space above the fat is kept full of steam. When boiling properly steam is turned off from the side pipe near the top of the cask and the boil is continued for from twelve to twenty-four hours. At the end of this period almost all of the remainder of the glycerin will have been extracted from the fat, which should now show 97 or 98 per cent of free fatty acids. Now add through the funnel about 0.05 per cent (or $\frac{1}{4}$ pound to 500 pounds of stock) of barium carbonate mixed with a little water, and after waiting fifteen or twenty minutes for it to be well boiled through, take a sample from the cock on the side, and if the water in the cask is neutral to methyl orange (that is, no longer appears red when a few drops of methyl orange are added to it) the steam may be turned off and the water allowed to settle out, which will take from one-half to two hours. If the first amount of barium carbonate added is not sufficient to make the water neutral to methyl orange, enough more to make it neutral must be added before shutting off steam, otherwise the fatty acids will discolor. The exact amount of barium carbonate required depends on the amount of the saponifying agent originally used and can be determined by trial as above. About one-tenth as much barium carbonate as of the saponifying agent will be needed provided no sulphuric acid has been added to the tank. The fatty acids will not now be affected by the atmosphere and may be drawn off and used at once or stored in wood until wanted. The water is left in the cask for the next boil, and if necessary enough water must be added to bring it up to what has been found the proper amount with which to start the boil.

Further Considerations.—The amount of the saponifying agent to be used is as follows: $\frac{1}{2}$ to $\frac{3}{4}$ per cent on prime, or No. 1, tallow, mutton tallow, yellow or white cottonseed-oil, cocoanut and palm-kernel oil, white grease, olive-oil, corn-oil, lard, good stearin, and good stock generally; $\frac{3}{4}$ to 1 per cent on off Nos. 1 and 2 tallow, very off cottonseed-oil, house grease, and similar stocks; 1 to 2 per cent on the worst grades of No. 2 tallow, yellow and brown greases.

The length of time of boiling on the first and second waters depends on the amount of the saponifying agent used, the quality of the fat, and the degree of decomposition desired.

Table XXVIII will give an idea of the results that can be obtained by varying the amount of the saponifying agent and the time of boiling with different fats.

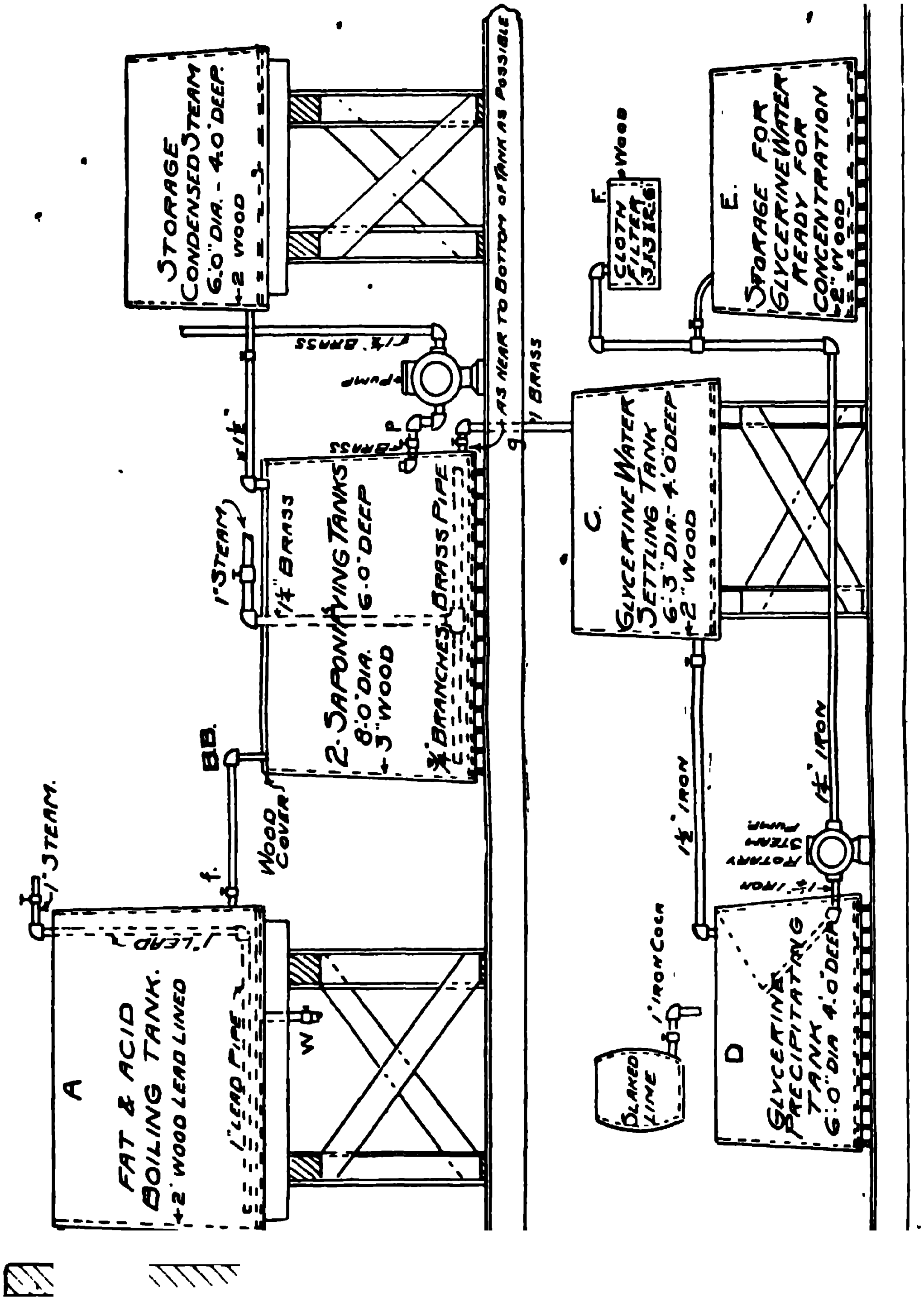

Storage Condensed Steam 6'0" Dia. - 4.0' Deep +2" Wood
1/4" Brass
Pump
P
1/4" Brass
As near to bottom of tank as possible
1" Brass
1 1/4"
1" Steam
1 1/4" Brass
2 Saponifying Tanks 8'0" Dia. 6'0" Deep +3" Wood
3/4" Branches Brass Pipe
BB
Wood Cover
f
1" Steam
A
Fat & Acid Boiling Tank +2" Wood Lead Lined
1" Lead
1" Lead Pipe
W
F
Cloth Filter 3'3"x2'6" Wood
E
Storage for Glycerine Water Ready for Concentration +2" Wood
C
Glycerine Water Settling Tank 6'3" Dia. 4'0" Deep +2" Wood
1 1/4" Iron
1 1/4" Iron
Rotary Steam Pump
1 1/4" Iron
1" Iron Cock
Slaked Lime
D
Glycerine Precipitating Tank 6'0" Dia 4'0" Deep

FIG. 174.—Apparatus for Saponifying 50,000 Pounds of Stock per Week. Twitchell process.

It must be remembered that the smaller the amount of the saponifying agent which can be used and give results the better will be the final color of the fatty acids.

In some cases (when a good color of fatty acids is of more importance than a high yield of glycerin) a high degree of decomposition may not be desired. In such cases the first boiling may be shortened and the second used only to treat the fatty acids with barium carbonate.

The proportion of water to fat and the progress of saponification can be very conveniently determined at any time by drawing off a sample from the side cock into a 100-c.c. graduated cylinder. Upon letting this stand a few minutes in a warm place, the proportion of water to fat can be read off and a sample of the fat taken from the top and tested for free fatty acids and a sample of the water taken from the bottom with a pipette and tested for free acid, using methyl orange as indicator. This water should always show an acid reaction and will show usually from 0.1 to 0.2 per cent H_2SO_4 at early stages of the boil and 0.04 or 0.05 per cent during the second boil.

If at any time during the boil the free acid in the water should be found to be less than 0.04 per cent of the water a little 60° Bé. sulphuric acid should be added, about 0.1 per cent of the weight of the fat being generally sufficient. If at any time during the boil the saponification is not progressing at the normal rate of speed the addition of 0.1 or 0.2 per cent of 60° Bé. sulphuric acid will generally result in effecting a return to the normal rate.

After a few runs have been made in the plant it will seldom be necessary to add any acid; but during the first 3 or 5 runs, or until the wood of the containers has been well boiled out, it is generally necessary to add 0.1 or 0.2 per cent of 60° Bé. sulphuric acid during the boil. If the acid content of the water should be found to be as high as 0.5 to 0.6 per cent free H_2SO_4 it would indicate that there had been a poor separation of waste acid from the fat on the preliminary acid wash and that too much acid had been drawn off with the fat. On account of the action of the wood on the saponifying agent the first three or four runs will never be as good both as to rate or degree of saponification and color as the latter runs. About four or five runs are generally necessary to thoroughly wash out the wood containers, so that the fifth or sixth run should be normal and representative of average results. It will take also several runs for the operator to thoroughly familiarize himself with the details of the

TABLE XXVIII.—COMPARATIVE RESULTS WITH THE TWITCHELL PROCESS.

		Prime Mutton Tallow.	Australian Tallow.	South Am'c'n Tallow.	Tallow.	Palm-oil.	Palm-oil.	Cocoa-nut-oil.	Palm-kernel Oil.	Palm-kernel Oil.	Olive-oil.	Olive-oil Foots.	Cotton seed-oil.	Linseed-oil.	Garbage Grease.
Free fatty acid in fat, per cent.		.6	3.0	4.5	2.57	19.8	19.8	1.8	8.9	8.9	4.9	40	0	2.3	40.8
Strength of spent acid water from acid boil, degrees Bé. .		8	11	10	8	8	8	8	9	10	6.7	18	17	12½	8
Amount of reagent used, per cent.		½	¾	1	3	½	1½	½	½	¾	8/10	1	½	¾	2
	Hrs.														
Percentage decomposition after boiling various lengths of time	3	4.8	......	......	58.7	33.0	53.0	......	......	......	......	......	......	......	73.0
	6	......	......	......	86.6		......	......	......	......	......	......	......	......	83.3
	7	......	......	......	89.8										
	9	......	......	......	......	......	......	......	......	......	......	......	......	......	87.9
	12	......	......	......	......		......	......	......	......	......	......	......	......	88.9
	16	......	......	......	......	......	94.8	73.1							
	18	67.1	......	......	......	77.6	......	......	......	......	......	......	47.3		
	24	80.2	81.2	91.3	......	85.2	......	85.5	84.0	87.1	92.0	92.5	71.0	85.8	
	30														
	36	......	......	......	......	......	......	......	......	......	......	......	89.9		
	40	91.8													
	48														
Ratio of fat to water at end of first boil		......	13:7	1:1	100:76	......	......	......	122:78	1:1	1:1	10:8	......	......	2:1
Strength of glycerin-water, degrees		......	6	3½	......	......	......	......	5	4	3	2			
	Hrs.														
Percentage of decomposition at different stages of the second boil	4	......	......	......	97.0										
	8	94.9	......	......	......	......	98.1	......	......	......	......	......	......	......	93.1
	12	......	......	......	......	......	......	......	......	......	......	......	95.7	......	96.7
	16	......	......	......	......	91.9	......	94.9	......	......	......	......	......	......	97.0
	20														
	24	......	94.2	99.1	......	95.2	......	......	93.1	98.0	96.0	96.1	......	9[illegible].4	
Total time of boiling	...	48	48	48	11	48	24	40	48	48	48	48	44	48	24
Usual time of first and second boils	1st	36	24	18	7	36	12	24	24	24	20	20	36	......	12
	2d	12	12	12	4	12	8	16	16	12	20	20	12	......	12

process and the apparatus so as to get the best results. Therefore, in experimenting with this process a number of runs—at least a half dozen—should be contemplated at the start.

To the glycerin-water drawn off at the end of the first boil (after settling and skimming off traces of fat) enough milk of lime is added to make it just alkaline, that is to turn red litmus paper blue. This generally requires about ¼ per cent of lime. The lime sludge is easily separated by settling or filtration, after which the glycerin-liquor is ready to be evaporated to crude glycerin by any of the usual methods.

When much lime has been used to neutralize an excess of sulphuric acid, it is preferable to evaporate the glycerin-water in two stages; first to 15° or 20° Bé. (45–60 per cent of glycerin). After this the calcium-sulphate is allowed to settle out and the clear liquor is concentrated to 28° Bé., equivalent to 1.24 specific gravity (88–90 per cent of glycerin).

In a small apparatus such as the one just described, the steam must be dry to prevent too much water being introduced. If the steam is too wet, a bleeder, or separator, must be put on the supply-pipe to keep the water of condensation out of the tank.

Factory Installations of the Twitchell Process.—Attention is directed to reproductions of plans of factory equipment for carrying out the Twitchell process on an industrial scale, shown in Figs. 174, 175, and 176. In Fig. 174 is shown apparatus necessary for effecting the decomposition of 50,000 pounds of stock per week. Referring to Fig. 174, about 10,000 pounds of fat are brought into tank *A* and boiled on sulphuric acid in the manner described in the operation of the experimental plant.

After settling and drawing off the acid water through pipe *w* 8000 to 9000 pounds of fat are run through pipe *f* into one of the saponifying tanks *B* and boiled with water and the saponifying agent as already described.

With the larger apparatus there will be much less condensation of steam in proportion to the charge and therefore about half as much water as fat may be added in the first place.

At the end of the first boil the glycerin-water is drawn through pipe *g* at the bottom of the tank into the settling-tank *C*, where

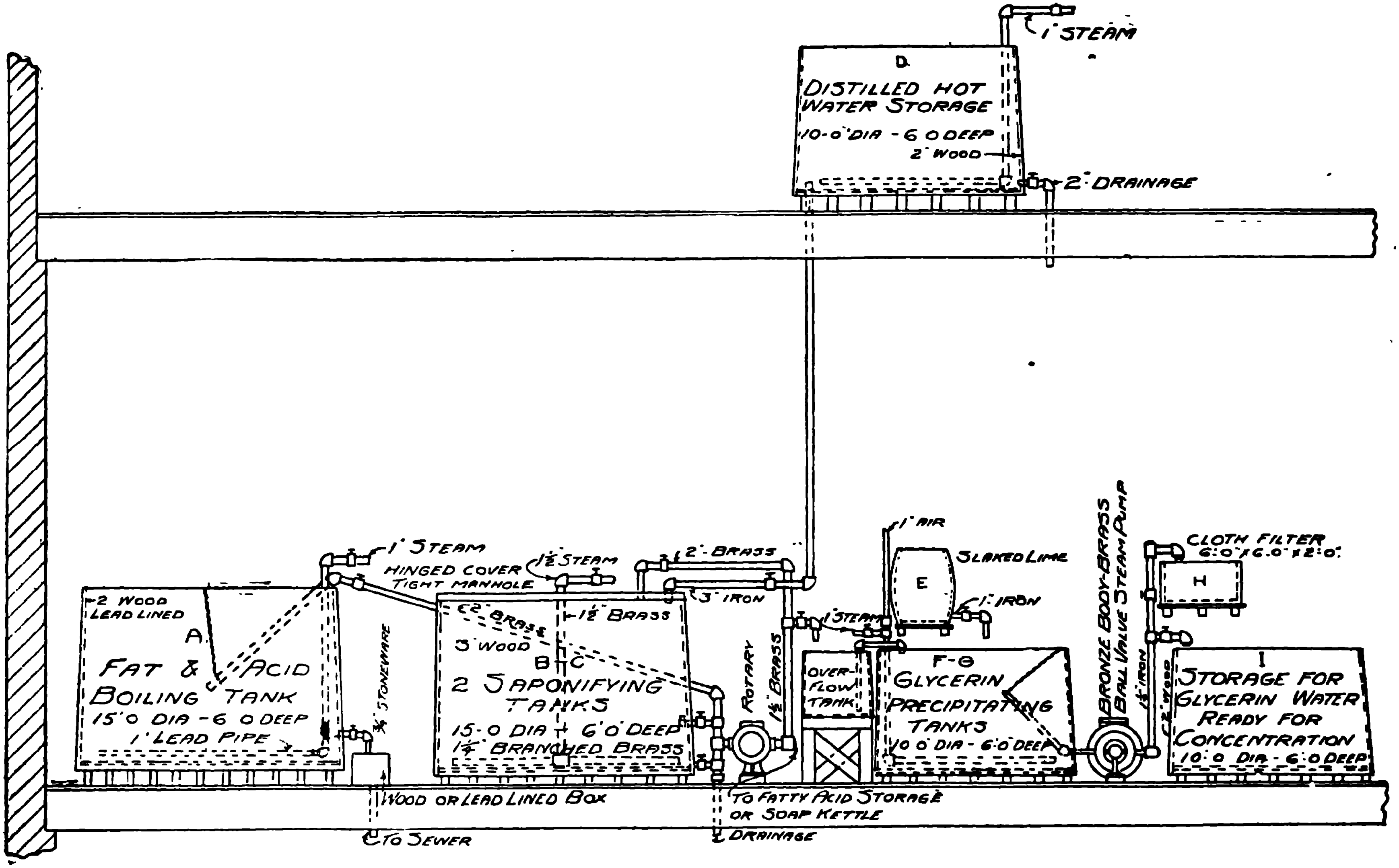


FIG. 175.—Apparatus for Saponifying 60,000 Pounds of Stock in One Operation All on One Floor. Twitchell process.

any fat which may have been carried with the water can be skimmed off.

From tank *C* the glycerin-water is drawn into *D*, where it is neutralized with lime as described on page 487. After settling in *D* the clear liquor is pumped by means of the swinging joint into storage-tank *E*. The sediment is allowed to remain in tank *D*. About once a week the swinging joint is lowered and the sediment is pumped, onto filter *F* and there washed. Or the contents of *D* may be pumped without settling, through a filter-press.

At the end of the second boil the fatty acids are pumped or drawn from the tank through pipe *p*. This pipe is placed at the proper height to draw off fat and leave the water in the tank. It should be turned upward inside the tank and have a try-cock in its side to test for water. Should water be found at the level of this pipe it will be necessary to draw off a little at *g* until the fat at *p* is perfectly clear.

Fig. 175 shows a plan of equipment necessary for saponifying 60,000 pounds of stock in one operation, with all apparatus located on the same floor. Fig. 176 shows a plan of equipment necessary for saponifying 10,000 pounds of stock in one operation, with the apparatus located on successive floors, whereby the use of pumps is avoided, all the material throughout the process being transferred by gravity.

Summary.—The chief desiderata to be obtained in working the Twitchell process are a complete saponification of the glyceride whatever may be its source and a light color of the fatty acids. The causes of failure to obtain a thorough saponification may arise from uncleanliness of the tanks, contact of contents of the tanks with iron, the use of hard or impure water, improper treatment during the preliminary wash, or the use of too little or too much water. Failure to obtain light-colored fatty acids may arise from the use of an excessive amount of the saponifying agent, the exposure of the contents of the tanks to air at any stage of the process before they are finally neutralized with barium carbonate, or the use of old or impure fat.

Paraffin. — Paraffin is a mixture of the solid hydro-carbons of the paraffin series and varies within somewhat wide limits in its hardness, melting-point, and color according to the source

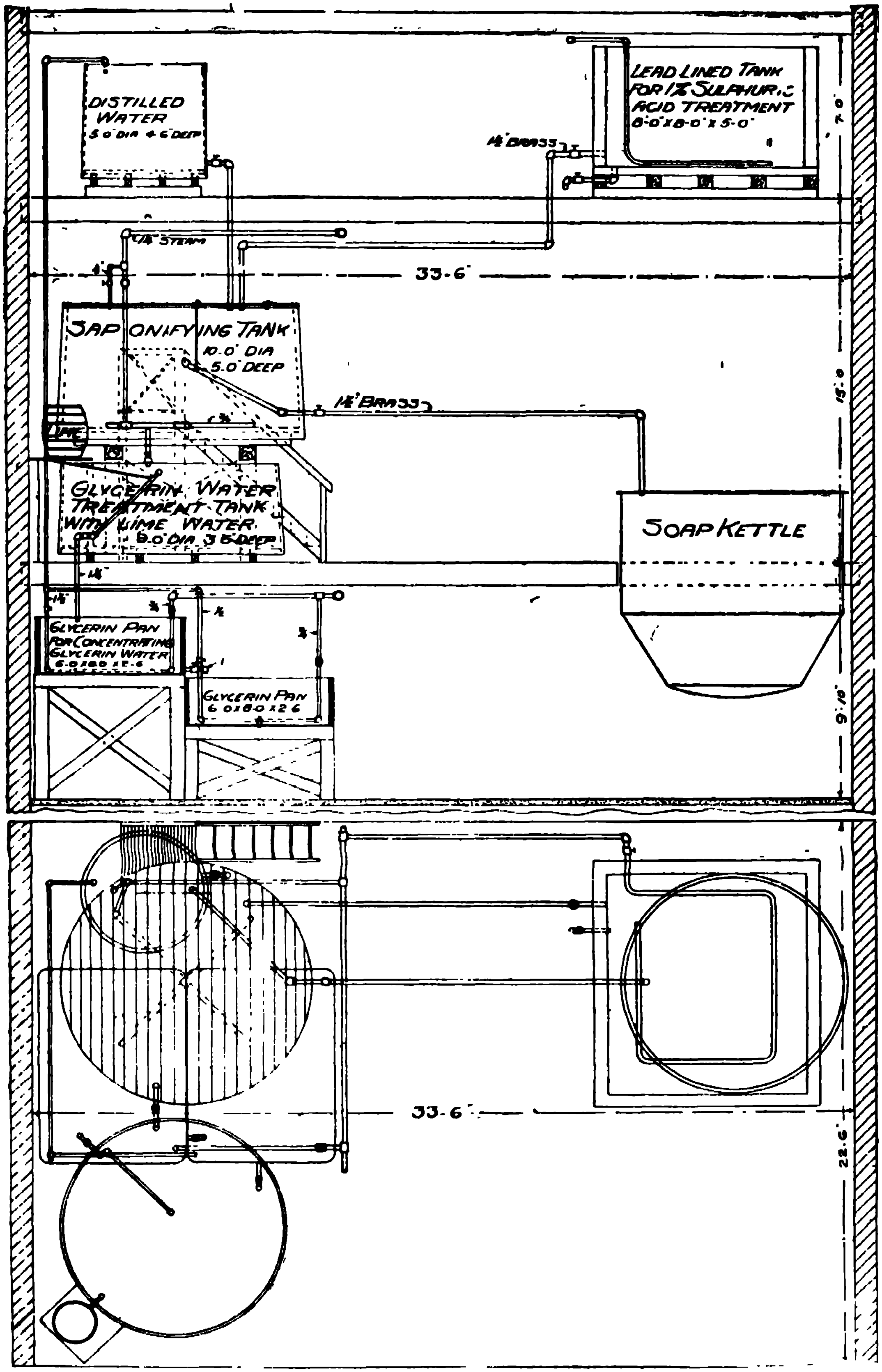

DISTILLED WATER 5-0 DIA 4-6 DEEP
LEAD LINED TANK FOR 1% SULPHURIC ACID TREATMENT 8-0 x 8-0 x 5-0
1¼ BRASS
1¼ STEAM
33-6
SAPONIFYING TANK 10-0 DIA 5-0 DEEP
1¼ BRASS
LIME
GLYCERIN WATER TREATMENT TANK WITH LIME WATER 8-0 DIA 3-6 DEEP
SOAP KETTLE
1¼
GLYCERIN PAN FOR CONCENTRATING GLYCERIN WATER 6-0 x 8-0 x 2-6
GLYCERIN PAN 6-0 x 8-0 x 2-6
7-0
15-0
9-10
33-6
22-6

FIG. 176.—Apparatus for Saponifying 10,000 Pounds of Stock in One Operation. Twitchell process The simplest arrangement of apparatus, avoiding pumps.

from which it has been derived and to the degree with which coloring-matter and softer bodies of similar composition have been separated. Refined hard paraffin is a resonant white or bluish-white translucent waxy solid, odorless, tasteless, of crystalline structure and, as indicated by its name, is characterized by chemical inertness. Softer varieties in proportion to the admixture of hydrocarbons of lower melting point depart correspondingly from the properties characteristic of the harder varieties. Ether, benzene, all light hydrocarbon oils, essential and fatty oils, and carbon disulphide dissolve it readily; it is sparingly soluble in hot alcohol, but separates on cooling. At a temperature below its melting-point it becomes plastic, which disadvantage to its use alone as candle-stock is overcome by the addition of varying percentages (5 per cent and more) of stearic acid, according to the hardness of the paraffin. When exposed under slight pressure for some time at a temperature below its melting-point, a change in molecular structure is undergone whereby paraffin becomes transparent; on change of temperature, or on being struck, its original translucent appearance returns.

The paraffin candle has grown greatly in favor since it was introduced; this is chiefly due to its greater illuminating power and the absence of the pungent and disagreeable odor that characterizes the old stearin candle when blown out. It has, however, the drawback of a somewhat low melting point, so that it cannot support its own weight in warm weather. The incorporation of a percentage of stearic acid minimizes this objection, as stated.

Petroleum paraffin is offered in five different grades, melting respectively at 120°, 125°, 130°, 135°, and 140° Fahr. Paraffin obtained by distillation of shales melts at about 140° Fahr., while that obtained from ozokerite melts at 140° to 158° Fahr.; if paraffin from the last source melts at a temperature lower than 136° Fahr., it may be assumed that hydrocarbons of lower melting-point have been surreptitiously incorporated.

Petroleum paraffin is obtained from the residuum remaining on the distillation of crude petroleum after the benzine and burning oil distillates have been separated. The residuum is transferred to "tar-stills" and subjected to further distillation; the lighter portions are returned for redistillation with crude or treated separately. The heavier portions, paraffin-oil, are collected and purified, first with sulphuric acid and finally with caustic soda and water, whereupon the product thus purified is chilled by refrigeration, moulded into cakes, and subjected to cold pressure. The refined heavy oil expressed is available as a lubricant; the solid hydrocarbons remaining in the cake are again subjected to greater pressure at a higher temperature, viz., 70° Fahr. To obtain refined paraffin, the crude paraffin thus obtained is crystallized from petroleum ether and the cakes subjected to two pressings as before; the paraffin thus purified is melted and filtered through boneblack, whence refined paraffin wax results.

Paraffin obtained from shale or brown coal is contained in shale-oil resulting from the distillation of the shale; shale-oil is distilled after the same general procedure as followed for crude petroleum. It differs, however, from the latter in that it yields no product corresponding to vaseline.

The shale-oil industry dates from Young's patent in 1850 and continued to be the sole source of paraffin for many years. Petroleum paraffin rapidly displaced paraffin derived from shale-oil and drove the Scotch industry into practical desuetude.

Ozokerite, or earth wax, varies in consistency from a soft substance to material as hard as gypsum, and in color from light yellow to black. It is widely distributed, but the most productive sources are in Galicia, where refined ozokerite, known as ceresin, is obtained by distillation of the crude material with superheated steam; the distillate is treated according to methods followed in the preparation of paraffin from petroleum. The high melting-point of ceresin, or ozokerite paraffin, admirably adapts it to candle-manufacture.

The illuminating power of ceresin candles exceeds that of the best paraffin candles from other sources. They have a high melting-point, and do not bend or soften in a warm atmosphere; they burn with a dry cup, and are not so liable to gutter as ordinary transparent candles; they are entirely free from smell and not at all greasy to the touch and resemble beeswax.

The chief difficulty experienced in the beginning in the casting of paraffin candles arose from lack of precaution in regulating the temperature while casting. If the moulds are colder than the inflowing paraffin, it will solidify as it touches the metal and assume a streaked appearance. On the other hand, if the moulds are allowed to cool slowly the candles will shrink on to instead of from the sides of the mould and will be injured in extraction, if they can be extracted at all without being melted out. When the moulding is properly carried out and with smooth moulds, paraffin candles are obtained with remarkable lustre. High degree of lustre, combined with translucency, ready adaptability to ornament in shape and color, and brilliancy of light give paraffin preëminence over all candle-stock. The readiness, however, with which paraffin liquefies imparts a tendency to gutter to even the best paraffin candles. Its property of becoming plastic after exposure to a moderate degree of heat, independent of its melting-point, is counteracted by the addition of 5 to 15 per cent of stearic acid. Inferior varieties of paraffin candles, i.e., from paraffin of low melting-point, are very liable to "smoke" from too abundant supply of fuel to the flame, whereby its temperature is chilled below that of ignition.

Stearin.—Stearin, consisting of the solid glycerides of the stock from which it is expressed (thus there may be tallow stearin, lard stearin, palm stearin, cocoanut stearin, cottonseed-oil stearin), was at one time used in the manufacture of candles and was an improvement over the use of tallow in that the more combustible and fluid olein was separated.

This term came to be applied to the solid fatty acids and to candles made from them. Cocoanut stearin mixed with fatty acids constituted the first "composite" candle. Stearins from whatever source are not used in candle-manufacture. They are, in the first place, a valuable source of glycerin and when expressed from tallow of good quality find a more remunerative outlet for edible purposes. When expressed from palm-oil the "stearin" consists largely of palmitin.

Stearic Acid.—Stearic acid, also called "stearine," is known as "saponified" and "distilled" "stearine" according as it is prepared by the lime-saponification process, or by acid saponification with distillation. This distinction refers to origin rather than to any difference in the practical working of the material obtained from the two sources. "Distilled stearine" in composition may be distinguished from "saponified stearine" by the presence of a small amount of iso oleic acid, to which the increase in yield of fatty acids by the acid distillation process may be attributed. Stearic acid from both sources is mixed with more or less palmitic acid. Stearic acid is a hard white, somewhat crystalline solid, lustrous and firm in texture and not greasy to the touch. It dissolves readily in alcohol, ether, and petroleum ether, separating from the hot solutions on cooling in small needle-shaped crystals. "Saponified stearine" solidifies at 126° to 132° Fahr. (52.2° to 55.5° C.); "distilled stearine" at 118° to 126° Fahr. (47.8° to 52.2° C.). Commercial stearic acid when melted and cooled slowly should show a close-grained crystalline structure and when broken into fragments should give a characteristic "snap." As "stearine," i.e., commercial stearic acid, always contains varying quantities of palmitic acid, the value of a given sample depends upon the percentage of stearic acid contained therein. By the determination of the melting and solidifying points the proportion of these two acids may be ascertained by reference to the following table.

TABLE XXIX.—MELTING AND SOLIDIFYING POINTS OF MIXTURES OF STEARIC AND PALMITIC ACIDS.

Stearic Acid, Per Cent.	Palmitic Acid, Per Cent.	Melting point.		Solidifying-point.	
		° C.	° F.	° C.	° F.
100	0	69.2	156.5		
90	10	67.2	153	62.5	144
80	20	65.3	149.5	60.3	140
70	30	62.9	145	59.3	138.5
60	40	60.3	140.5	56.5	133
50	50	56.6	133.5	55	131
40	60	56.3	133	54.5	129.5
32.5	67.5	55.2	131.5	54	129
30	70	55.1	131	54	129
20	80	57.5	135	53.8	128.5
10	90	60.1	140	54.5	129.5
0	100	62.0	143.5		

Stearic-acid candles have a greater illuminating power than those made from glycerides, are harder and possess none of the disadvantages arising from the decomposition of the glyceride during the combustion of the candle. They are inferior, however, in illuminating power to those made from paraffin.

Mixtures of Stearic Acid and Paraffin.—In practical work calculation of the proportion of ingredients is based upon the predominant material; thus a 50 per cent paraffin candle means 100 parts of stearic acid and 50 parts of paraffin. In tables of melting-points of mixtures of commercial stearic acid and paraffin, this proportion is not so indicated. Such a table should be compiled by the works chemist from tests applied to the materials used and their common mixtures. In the following tables are compiled melting-points of mixtures in various proportions of commercial stearic acid and paraffin of different melting-points. Great lack of uniformity of results in the determination of melting points arises from the use of different methods and different manipulation of the same method. As shown by these tables a mixture of paraffin and "stearine" melts at a lower temperature than would be calculated from the melting-points of the two materials. The addition of "stearine" to paraffin causes candles made from the latter material to become opaque and whiter, while at the same time it corrects the tendency of paraffin to soften at a temperature below its melting point.

Miscellaneous Candle-stock.—The principal materials, other than commercial stearic acid and paraffin, used in candle-manufacture comprise beeswax, which in primitive times formed the illuminant of the wealthy classes but now is of limited use and almost exclusively confined to candles for religious purposes; spermaceti, used exclusively in the manufacture of candles designed as the standard of photometrical measurement; cerasin, the refined product of ozokerite; and of minor importance, Japan wax, carnauba wax, Chinese wax, etc.

The waxes are characterized by being solid bodies having a peculiar semi-glistening lustre, generally denominated as waxy. The texture varies; some are homogeneous, like Japan wax and carnauba wax; beeswax is granular in appearance, while spermaceti and Chinese insect wax are decidedly crystalline; all are sufficiently soft to be cut with the finger-nail, but hard enough to break when bent or struck. They melt usually from 125° to 140° Fahr., and are all lighter than water. They are of complex composition and characterized in the main by being salts of fatty acids with mono- or di-atomic alcohols and hence yield no glycerin. As wax cannot

TABLE XXX.—MELTING-POINTS OF CANDLE MATERIAL FROM MIXED PARAFFIN (SCOTCH) AND "STEARINE" (I. I. REDWOOD).

A.

Paraffin.		" Stearine."		Mixture.
Per Cent.	Melting-point.	Per Cent.	Melting-point.	Melting-point.
	° F.		° F.	° F.
90	102	10	121	100
80	"	20	"	98.50
70	"	30	"	100.0
60	"	40	"	104.50
50	"	50	"	110.50
40	"	60	"	111.0
30	"	70	"	113.50
20	"	80	"	117.50
10	"	90	"	119.0

B.

Paraffin.		Stearine."		Mixture
Per Cent.	Melting-point.	Per Cent.	Melting-point.	Melting-point.
	° F.		° F.	° F.
90	120	10	123	118
80	"	20	"	116.50
70	"	30	"	114
60	"	40	"	112
50	"	50	"	110
40	"	60	"	109
30	"	70	"	113
20	"	80	"	118.50
10	"	90	"	119.50
	"			

C.

Paraffin.		'Stearine."		Mixture.
Per Cent.	Melting-point.	Per Cent.	Melting-point.	Melting-point.
	° F.		° F.	° F.
90	120.25	10	129.75	118.50
80	"	20	"	116.75
70	"	30	"	114.50
60	"	40	"	112.25
50	"	50	"	113
40	"	60	"	118.75
30	"	70	"	122
20	"	80	"	124.50
10	"	90	"	127

D.

Paraffin.		"Stearine."		Mixture.
Per Cent.	Melting-point.	Per Cent.	Melting-point.	Melting-point.
	° F.		° F.	° F.
90	125	10	121	123
80	"	20	"	121
70	"	30	"	119
60	"	40	"	117.50
50	"	50	"	114
40	"	60	"	111
30	"	70	"	107
20	"	80	"	114
10	"	90	"	117

E.

Paraffin.		"Stearine."		Mixture.
Per Cent.	Melting-point.	Per Cent.	Melting-point.	Melting-point.
	° F.		° F.	° F.
90	130	10	121	128
80	"	20	"	125.50
70	"	30	"	123
60	"	40	"	121
50	"	50	"	118.50
40	"	60	"	114
30	"	70	"	109
20	"	80	"	115.50
10	"	90	"	118

F.

Paraffin.		"Stearine."		Mixture
Per Cent.	Melting-point.	Per Cent.	Melting-point.	Melting-point.
	° F.		° F.	° F.
90	132.50	10	129.75	130.50
80	"	20	"	128.50
70	"	30	"	126.50
60	"	40	"	124.25
50	"	50	"	121.0
40	"	60	"	117.75
30	"	70	"	119.50
20	"	80	"	125.25
10	"	90	"	127.50

be moulded, candles made from it alone must be "dipped." Beeswax makes excellent candle stock, being intermediate in illuminating power between stearic acid and paraffin. The high price of beeswax gives constant encouragement to adulteration. Stearic acid, paraffin, cerasin, rosin Japan wax, carnauba wax, and stearin are most commonly used for this purpose. Waxes, owing to their high melting-point, serve to harden candles, the lustre also being increased. Pure spermaceti, being very brittle, cannot be employed alone for candles, but is mixed with beeswax, stearic acid, or paraffin. In the directions for manufacturing the standard sperm candle it is prescribed that the best air-bleached beeswax, melting at or about 144° Fahr., and no other material, shall be used for this purpose, and that the proportion of beeswax to spermaceti shall be not less than 3 per cent nor more than 4½ per cent.

Bleaching Beeswax and "Stearine."—The natural method of bleaching, which still remains the best and of all others alone preserves the natural characteristic aroma in beeswax, lies under the single disability of taking up a considerable time in its performance. There is, it is true, a possibility of shortening the process by the employment of ozone for the artificial enrichment of the bleaching atmosphere, but this has only an insignificant influence unless care is taken to preserve the wax in a certain condition of moisture. To supply this ozone we may proceed by atomizing oil of turpentine or by making use of the electric current. Still, even under these circumstances the bleaching process may be protracted for some weeks, or even months; so that the advantages of a chemical process capable of rendering the wax perfectly white in twenty-four hours cannot be overlooked. In practice pure beeswax is rarely bleached. It is customary to add to yellow wax, prior to bleaching, from 3 to 5 per cent of tallow for the purpose of counteracting the natural brittleness of pure bleached wax; moreover, in the presence of a small quantity of tallow, bleaching ensues more rapidly, and without the tallow it is difficult to obtain a product entirely white. The action of tallow in bleaching is attributed to the presence of oleic acid, which being easily oxidizable aids the combustion of the organic coloring-matter of the wax. Oil of turpentine added in small quantities acts in the same manner. Other processes may be employed for decoloring wax, e.g., bleaching with animal charcoal. The wax is kept in a fused state in contact with the black, and the white wax is obtained by filtration. Permanganate of potash or bichromate of potash may also be employed in acid solution with good results, and even oxygenated water.

Reducing agents, such as sulphurous acid, sulphites, hydrosulphites, etc., do not act on the coloring-matters of wax. Chlorine bleaches waxes, but the chlorine is absorbed as bromine and iodine are, and thus the composition of the wax is greatly modified.

From a close investigation into the natural method of bleaching it appears* that the duration of the process is influenced by the following factors: The percentage of water in the wax, the moisture of the air, the surface of the wax, the temperature, and the light. Perfectly dry wax takes twice as long to bleach as that containing from 2 to 5 per cent of water, while, on the other hand, air laden with moisture will only bleach four times more slowly than if quite dry. The most favorable temperature is 35° C., though the operation can be effected at as low as 20° C. Diffused sunlight (full daylight) is almost as effective as the direct rays of the sun. The most important factor, however, is the amount of exposed surface, which should be as large as possible. To this end the crude wax may be melted and run into laminæ of one-fifth to one-eighth of an inch in thickness, or, better still, made up into an emulsion with water at 60° to 80° C. in a Laval emulsifier and poured out into cold water, by which means it is obtained in the form of extremely small granules. It is also found that the presence of bleached wax in the crude wax expedites the bleaching of the latter considerably.

By the aid of the emulsifier it is claimed wax can be chemically bleached in ten minutes. To accomplish this the wax is emulsified with slightly alkaline water at 80° C., and after exposing the emulsion to the action of sodium hypochlorite for ten minutes a slight excess of hydrochloric acid is added, the wax being finally washed with warm water in the apparatus. The product is, however, inodorous and brittle.

Stearin behaves exactly like wax under the natural bleaching process, and the chemical method is also applicable to Japan and carnauba wax. In practical bleaching processes the loss or shrinkage in volume of the bleached wax is variable, according to the composition of the crude wax, and is of such an amount as to give rise to the custom in the candle trade of considering 14 ounces the weight of a pound of beeswax candles.

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* Buisine, Bull. Soc. Chim., p. 465, 1890.

The Candle.—A candle consists of a cylinder of solid, fusible, combustible matter surrounding and saturating an axis which is itself combustible and in virtue of the capillarity of its parts admits when ignited of the continual absorption and ascent of the surrounding matter, liquefied by its heat of combustion, where it is distilled; the gases thus formed and raised to the temperature of ignition burn, the incandescence of the carbon particles resulting from their decomposition providing illumination. In its structure is to be found vestiges of the *pine branch* which in primitive times, as a torch, served as the sole instrument of illumination; of the *link*, or rope strands, steeped in resin, tar, or pitch; of the *flambeau*, or core of hemp, soaked in resin and coated with beeswax, and of the later *dip* formed by coating strands of yarn by repeated dipping and cooling in molten tallow or wax. With the introduction of moulds by Sieur de Brez in the fifteenth century, the candle in exterior form as seen to-day first appeared. The nature of the combustible, its melting-point, viscidity, and burning power, the blending of suitable material from several sources, selection of the wick that in number of threads, substance, and structure is adapted to the combustible chosen, are practical considerations for the candlemaker.

Preparation of Wick.—Wicking is made from the best quality of cotton and carefully spun so that there will be no loose fibres or threads, but that the yarn will be uniform throughout. The cotton having been bleached by the spinner is spun into yarn, which to indicate its purpose is termed "wicking," and is then cut into hanks of uniform length, in which form it is delivered to the candlemaker. To facilitate combustion of the wick, and to counteract "smoking," the wicking is first pickled, which consists in immersing the hanks in a dilute solution of mineral salts for a period of twenty-four hours. The mineral salts used comprise borax, chloride or nitrate of potassium, ammonium chloride, or chloride sulphate or phosphate of ammonia. Pickling causes the wick to curve in burning, and at the same time to vitrify the ash. It also prevents the wicks from burning too rapidly and obviates the necessity for snuffers. After steeping, the hanks are placed in a centrifugal machine and the solution thoroughly expelled, after which, if necessary, they may be further dried in a drying-room.

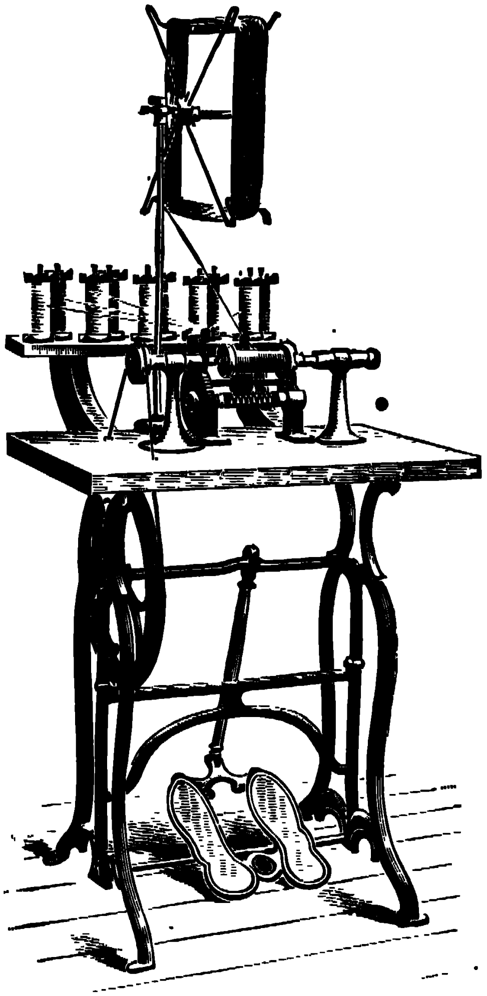

FIG. 177.—Spool winder.

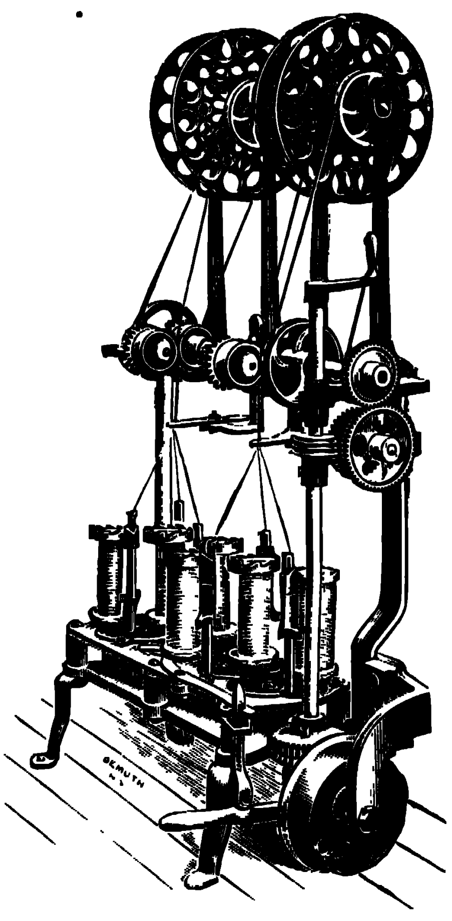

FIG. 178.—Wick-braider.

They are considered sufficiently dry when they present a characteristic crisp feel. The proportion of mineral matter retained by the wicking is extremely slight, but yet sufficient to materially affect the burning of the candle. The wicking is now ready to be wound on spools or bobbins, for which purpose a spool-winder, shown in Fig. 177, is used. The hank is placed on the reel and wound on the spool as shown. The same machine is used for gathering threads from any number of spools according to the size of the wick. Assuming that a wick of thirty threads is to be made, strands made of threads from ten spools are wound on one spool and three of these spools, bearing strands of ten threads each, are placed in the wick-braider shown in Fig. 178. The spool to receive the plaited or braided wick is inserted between the guards at the top of the machine and receives the braid formed by the automatic movement of the device carrying the spools at the base of the machine. The spool when filled with the wick thus made is ready to be inserted on the spindle of the candle-machine.

The Wick.—The size of the wick must be adjusted to the diameter of the candle and the fusibility of the material. The capillarity of the wick, i.e., the rate at which molten matter is drawn up from the gutter formed by the melting of the combustible and its absorption by the wick, must be such that the exact quantity of fluid combustible shall rise through it and be burned with a flame sufficiently large and hot to consume as much combustible as it melts. If the wick is too large, i.e., the capillarity too great in proportion to the diameter of the candle, no cup at the base of the wick is formed and guttering ensues. If the capillarity is too slight, the unmelted matter forming the rim of the candle does not melt regularly with the descent of the flame, thus producing equally unsatisfactory results. The more easily melted and combustible the material, the looser must be the plait of the wick; hence a plaited wick is unsuited for a tallow candle. By the size of wick is meant the number of strands or threads contained in it, and varies as stated according to the diameter of the candle and the fusibility of the material. The number of threads in each strand varies from 3 to 20. The size of the wick is indicated by the number of threads in each strand and the number of strands. Thus a 3 10 wick contains 30 threads in 3 strands of 10 threads each. The looseness of the plait is likewise determined by the same conditions and varies from 4½ to 18 per inch.

A candle of very fusible and combustible material should have a looser plait than one made of material of higher melting point and less ready combustion, with the number of threads depending upon the diameter of the candle. Thus the diameter of the candle and the fusibility of the material determine the size of the wick and the number of plaits per inch.

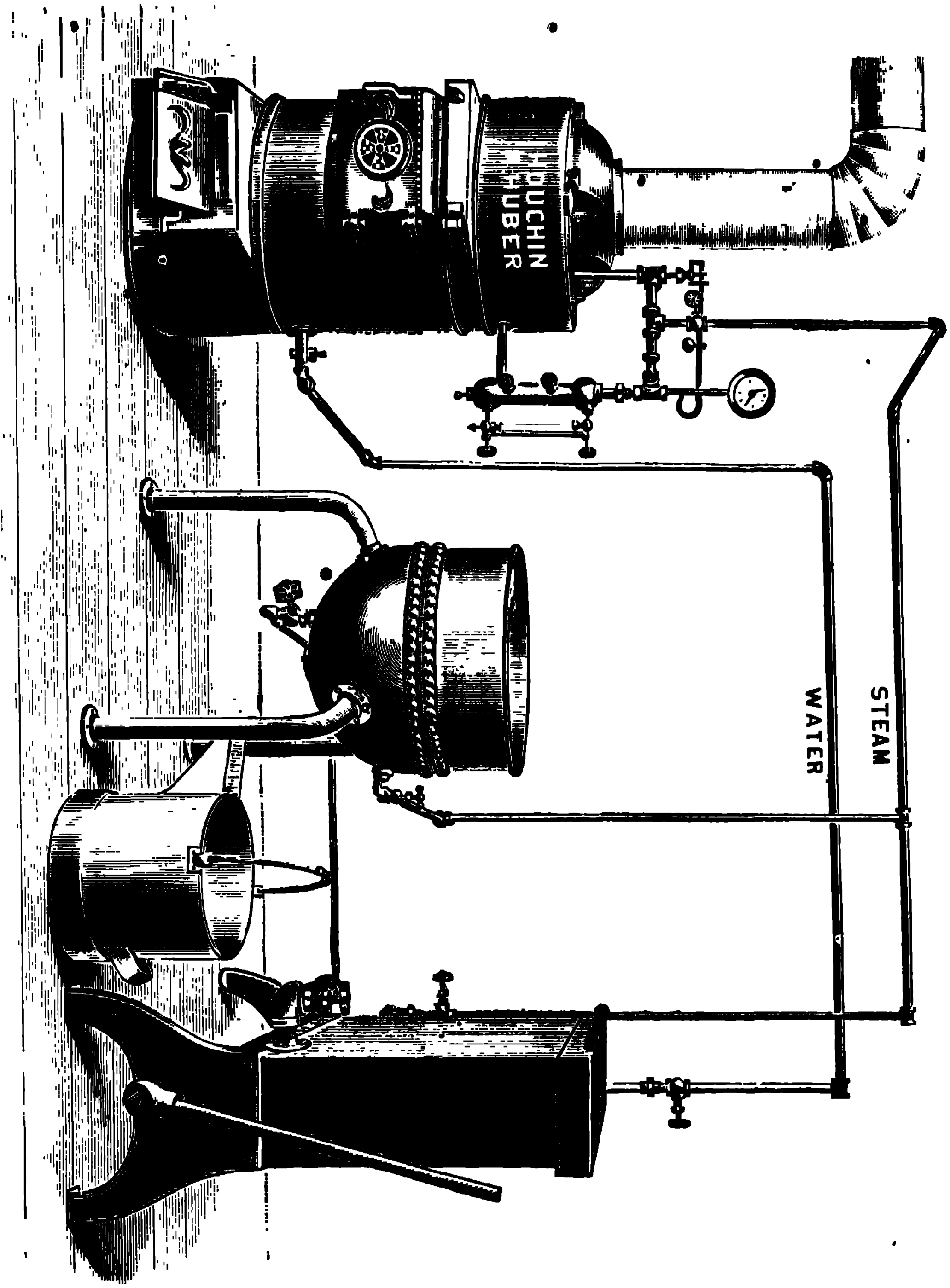

FIG. 179.—Elements of the Equipment of a Candle-plant.

The size of a candle is indicated by the number required to weigh a pound; and with a given number of candles per pound there is further variation in diameter and length. With large diameter there is corresponding reduction in length and vice versa. The plait in burning contracts unequally and bends outside of the zone of combustion, where in contact with oxygen of the air it is completely consumed.

Equipment of the Candle-plant.—The essential equipment of a candle-plant comprises a boiler to supply steam to melt the materials and to heat the moulds; a water-supply under pressure to cool the moulds; jacketed kettles in which to melt the materials; a wick-plaiting machine where the wick is not provided ready for use; a wick-winding machine for filling spools of the candle-machines; candle-makers' tools, such as a tin swimmer, tin pouring-bucket, steel cutter or scraper, a steel wick-knife and thermometer, to which may

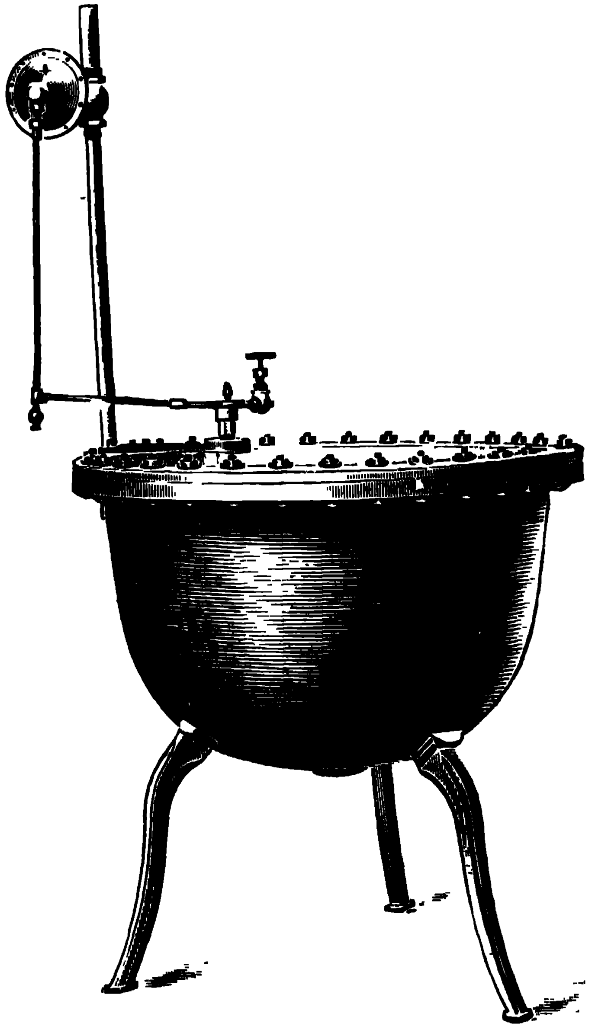

FIG. 180.—Melting-kettle, Aluminum-lined, with Automatic Temperature Regulator.

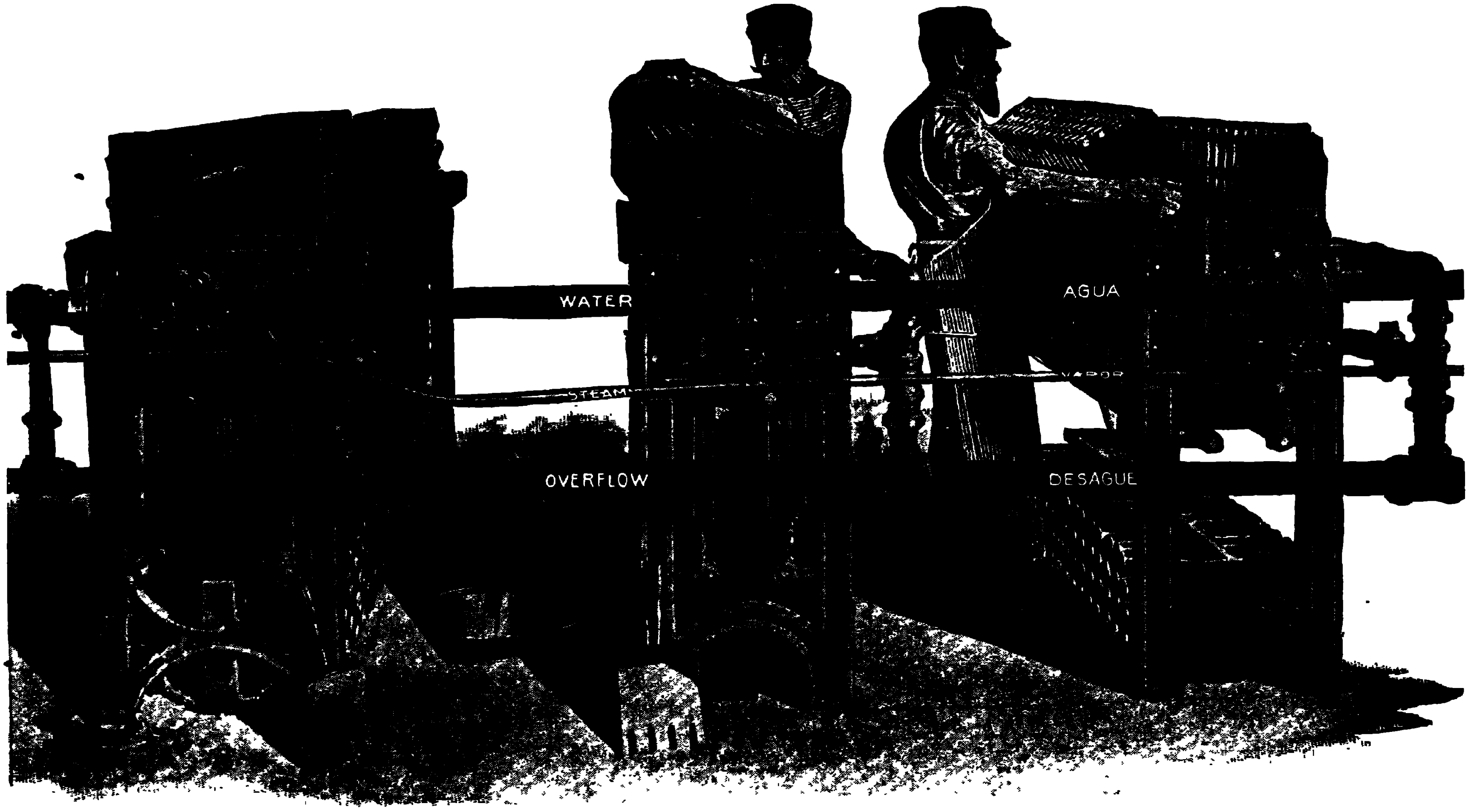

FIG. 181.—Candle-machines, Showing Connections and Manipulation.

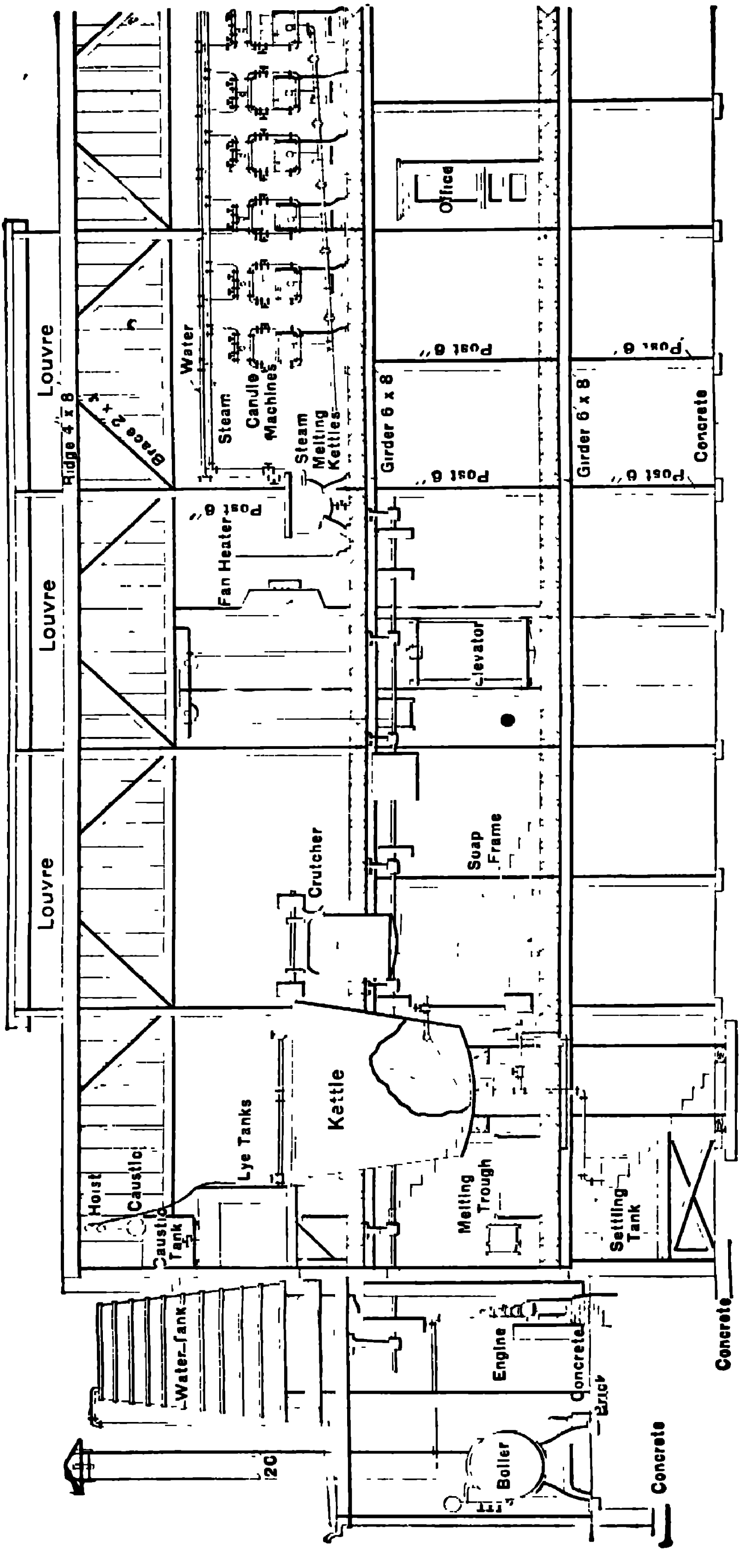

FIG. 182.—Combined Soap- and Candle-plant.

be added a foot- or steam-power butt-saw and steam stamping-machine, or a combination sawing-stamping-polishing machine; also pipe-cutting tools and extra fittings for repairs where the factory is remote from a market for such goods.

The essential apparatus of candle-manufacture are shown in Fig. 179, which is a view of a plant for large-size candles, showing steam-boiler, aluminum jacketed melting-kettle, and candle-machine, with suitable water and steam connections; also a tin pouring-bucket.

On pages 505 and 506 are shown methods of setting up, piping, and filling the candle-machines and of removing the racks of candles. These methods naturally vary with each establishment and for which no hard-and-fast rule can be made. Fig. 182 is of especial interest in that there is shown a plan of the actual equipment of a combined soap- and candle-plant.

Melting-kettle.—The melting-kettle may be heated either by steam or direct fire, but preferably by the former. All parts in contact with melted material should be non corrosive, so as to avoid discoloration of the candle-stock. For this purpose steam-jacketed kettles lined with aluminum give the best satisfaction, as this metal is light, durable, and not affected by the acids.

Paraffin may be melted in a kettle of aluminum or iron. When mixed with stearic acid in the preparation of a candle-stock the composite should be melted in wooden tubs, earthen crocks, tin vessels, porcelain or aluminum kettles. Iron will discolor the stock and lead-lined tubs will injure the burning quality of paraffin. A coil of iron or lead pipe, however, can be used for heating the tubs or kettles provided it is placed at the bottom and covered with enough water to keep the stock from coming in contact with the iron or lead of the coil. The stock can be heated on the water with impunity, but it should be well settled before use, in order that no water shall get into the moulds or the candles. The required kettle capacity of a candle-plant depends on the number and size of the moulding-machines used. An aluminum-lined melting-kettle is shown in Fig. 180.

Mixing Ingredients.—The paraffin-stearic acid for composite stock may be melted separately or in the same vessel. Very little agitation is required to combine them when they are melted separately. When melted in the same vessel, it is advisable to melt the paraffin first; but where the stock is melted on water, it is immaterial which ingredient is first melted, or whether both ingredients are put into the same vessel together to be melted or melted separately.

Coloring Candles.—In coloring moulded candles the dye is uniformly mixed with the melted stock; with wax candles made by dipping, the candle may be colored throughout by mixing the dye with the melted stock or by dipping the candle at intervals during formation, or once, at the end, into stock mixed with the dye. The dipped candle may thus be either colored uniformly or only upon the surface, or in variegated colors. Mineral colors are inadvisable owing to their interference with combustion. A great variety of aniline colors are available. As a rule the color in requisite amount is dissolved directly in a small portion of the melted stock and the solution strained into the main body of the material.

Stearic acid dissolves many aniline colors, although a few of them in limited quantities only. Its acid nature causes many of the colors to fade. The basic character of many of the anilines explains the readiness with which they are dissolved by stearic acid. The latter is heated to a little above the melting-point, 60° to 65° C., and the coloring added.

The quantity of the coloring-matter varies between 0.05 and 0.01 per cent of the weight of the stearic acid according to the depth of color desired. Care should be taken not to overheat the mass lest the color be decomposed.

Some of the colors are so sensitive to heat that freshly moulded candles in which they are used appear quite colorless, the color only appearing when the candles have become quite cold. The blue colors on warming also sometimes turn reddish, recovering the original color on cooling.

Some of the colors which are quite permanent on textiles prove decidedly fugitive in stearic acid, and when a mixture of yellow and brilliant green is used the latter fades more rapidly than the former, leaving ugly greenish-yellow colored candles.

Good results are obtained with the following:

Red.—Sudam IV., rhodamin, flexin, rose bengal, and fuchsin.

Yellow.—Chinolin yellow, auramin "lederin."

Green.—Victoria green, acid green, and a mixture of victoria blue and chinolin yellow.

Blue.—Victoria blue, methyl violet, and indulin.

The use of aniline colors in beeswax is very similar to that in stearic acid.

Paraffin and ceresin, being inert bodies, have different solvent action, but for the small quantity of dye employed there is no practical difference.

Candle-moulding Machine.—The modern candle-machine is a device for the more efficient utilization of the old tin-mould originally used for tallow candles. The principle of the original device, Fig. 183, persists with no modification from the date of its conception in the fifteenth century. Utilization of the wick, as of old, is continuous, the improved features of the modern machine being in the greater number of candles moulded at a single operation, from 24 to 360 and more according to their size, and the rapidity of operation. Although the development of the present machine through a period of four centuries has been slow, the ingenuity expended has been by no means slight. Its slow growth, with successive applications of ingenuity to its various parts, eliminates the element of individuality from the modern candle-machine (*vide* page 445), and leaves undisturbed the repute of Sieur de Brez, the originator of the candle-mould. This repute in turn is not unassailable, for the hands shaping the plastic material of the primitive dip was the first mould.

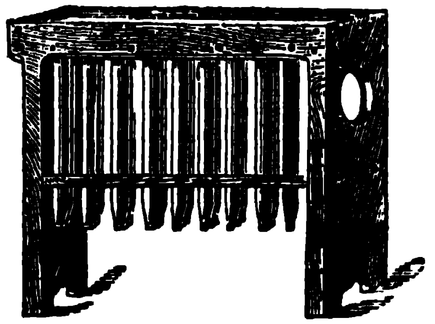

FIG. 183.—Hand Candle-mould.

The candle-machine consists essentially of a series of vertical cylinders with the tops terminating in the bottom of a shallow tray. Each cylinder, or mould, contains a hollow piston the head of which moulds the top or tip of the candle, the cylinder and movable piston-head, therefore, forming the candle-mould (Fig. 184). Through the movable piston is inserted the wick wound on spools inserted horizontally on spindles below the board forming the lower attachment of the piston, and to which motion in a perpendicular direction is transmitted by means of a rack and pinion. The series of vertical cylinders, varying in number from 24 to 360, are surrounded by a chamber through which they may be said to project and fitting tightly at the top and bottom. This chamber is suitably connected with steam and water for the purpose of heating the moulds preparatory to the introduction of melted candle-stock and for cooling the moulds when filled.

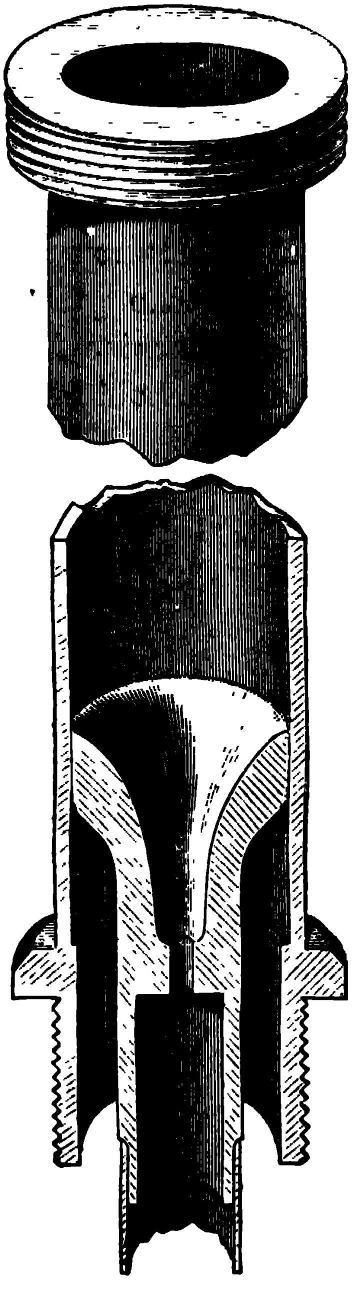

FIG. 184.—Single Candle-mould.

Construction.—Referring to the candle-machine shown in Fig. 185, the machine consists of a stout iron frame *AA'*, supporting a metallic box or chest *B*, containing in this instance 240 vertical candle-moulds whose upper ends open directly into a pair of horizontal channels, into which the melted stock is poured. These moulds are traversed by tubular pistons *C*, attached to a common follower *D*, which is raised and lowered by racks *EE'*, the power being applied at crank *G*. The upper ends of the pistons are conical, to impart the usual tapering tip to the candles, and each piston has a wick passed up through it, the various wicks being wound upon spools, or bobbins, *H*, situated at the base of the machine. *J* and *K* are removable racks in which the moulded candles are clamped. *M* is the overflow for cooling-water. The system of piping to provide steam and water for heating and cooling in machines of this type is shown in Fig. 181.

Referring to the 96-mould candle-machine shown in Fig. 187, a difference in construction from the type shown in Fig. 185 may be seen. In this machine a double-mould chest is used which is a feature of practical value; in event of leakage of the mould-chest the entire machine need not be shut down, this feature permitting one side or one half of the machine to be used continuously no matter what may occur to any mould or part of the machine.

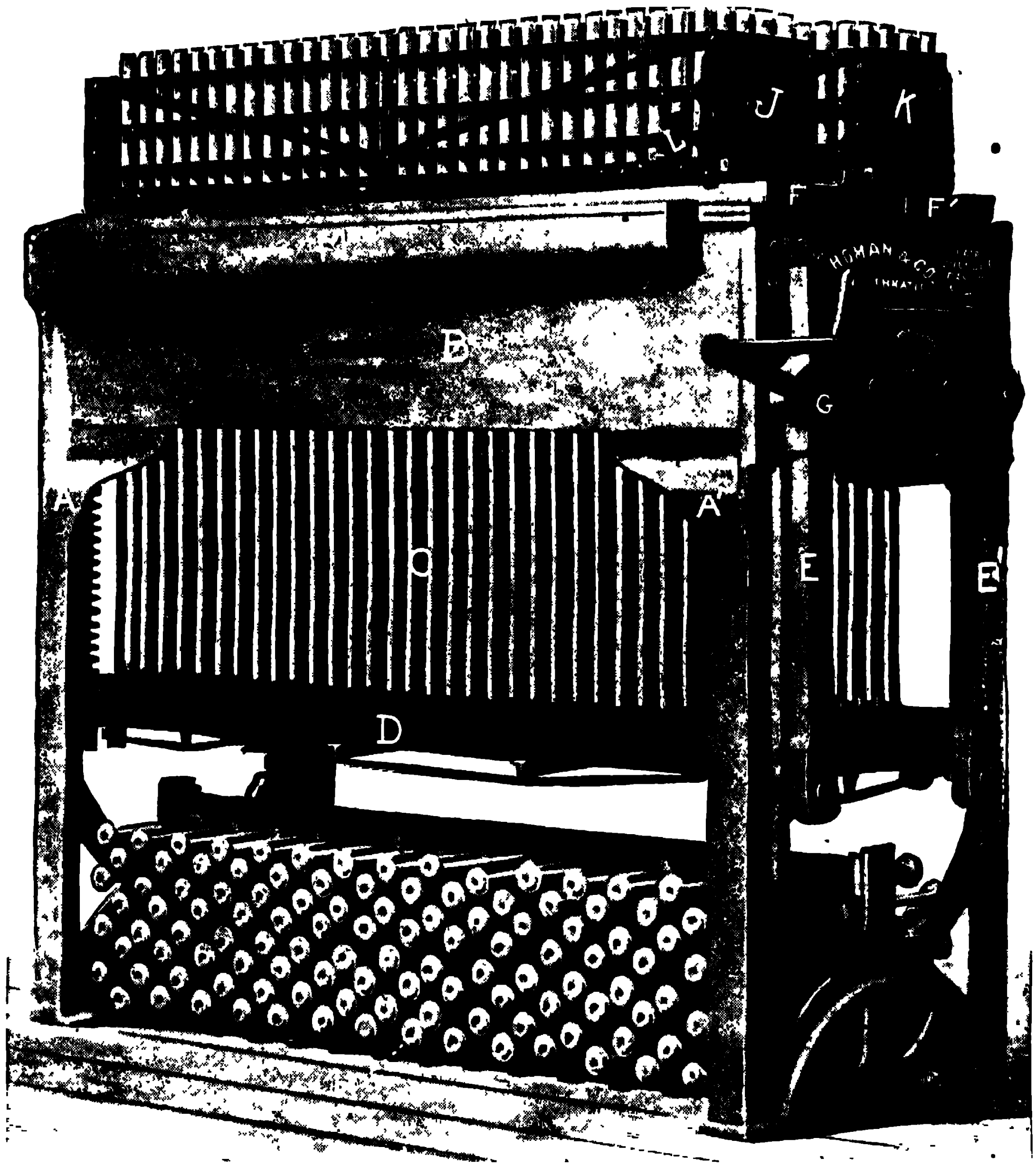

Fig. 185.—240-mould Candle-machine.

Machines of this type are shown set up in series in Figs. 182 and 186, the arrangement of the feed water and steam-pipes and of the common discharge-pipe for hot water being determined by practical considerations of location and space available. A 360-mould machine of this type is also shown in Fig. 188. The construction of the mould-box in this manner provides a two-sided or double machine which permits either side of the driving-board operating the pistons to be raised at pleasure, giving at once the effect of two separate machines and permitting repairs to be made to either side without disturbing the working possibilities of the other.

The mould-box in the single-box machines is a stout casting with a trough at each side to receive the overflow, the box and troughs forming one casting. In the double-box machines the boxes are cast separately and the overflow discharged through a pipe. The mould-box varies in size according to the length and number of moulds.

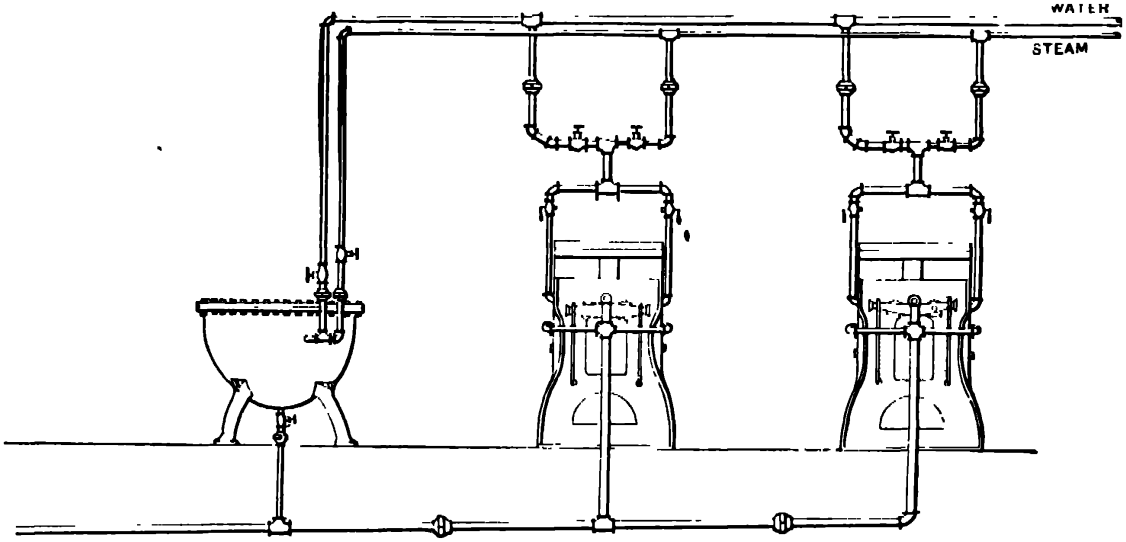

FIG. 186.—Steam and Water Connections of Candle-machines. Double box type.

The moulds constitute the most vital part of a candle-machine. They are arranged perpendicularly in the mould-box in two sections and in parallel rows. In the single-box machines the head of each mould is screwed into the tray that covers the mould-box and the lower end is secured by a shoulder and washer on the inside and a nut on the outside.

In the double-box machines the mould is attached to the top and bottom plates of the box in such a manner as to also serve as a stay-bolt. The moulds are cast of a high-grade tin and their interior is given the finest possible finish. A polish is thereby imparted to the candles, doing away with the necessity of using a polishing-machine.

The mould illustrated in Fig. 184 may be easily removed in the following manner: Remove the piston by loosening the bolts of the lower rack. After taking off the nut from the bottom, insert in the top of the mould a conical piece of wood about 15 inches long having

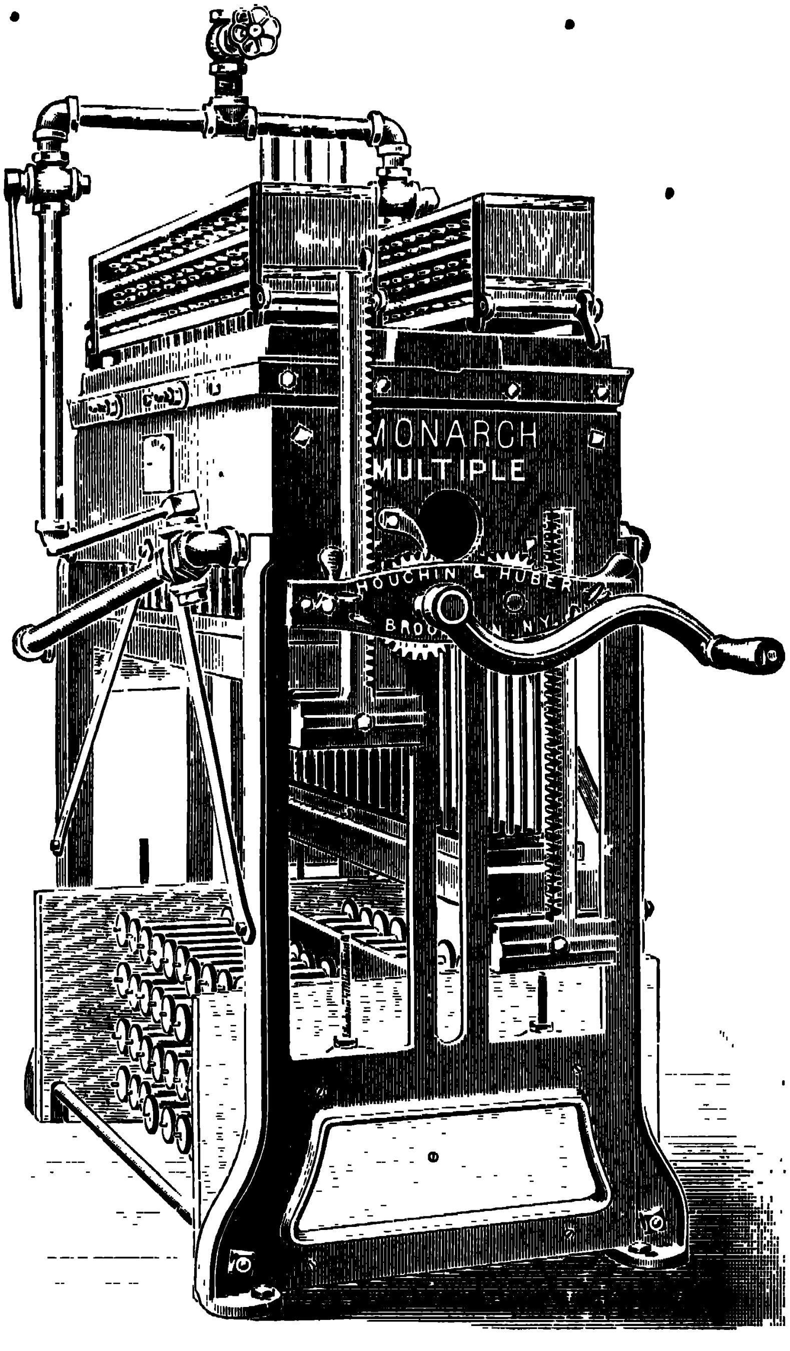

FIG. 187.—96-mould Combination Candle-machine.

an iron or wooden pin piercing it at the large end to serve as a handle. Tap the end of this tool until it wedges sufficiently tight to permit of unscrewing the mould. The new mould can be fitted by the reverse operation. Before inserting the piston, clean the mould thoroughly by means of a rod covered with a clean soft cloth, otherwise the piston-tip may scratch the smooth interior surface of the mould.

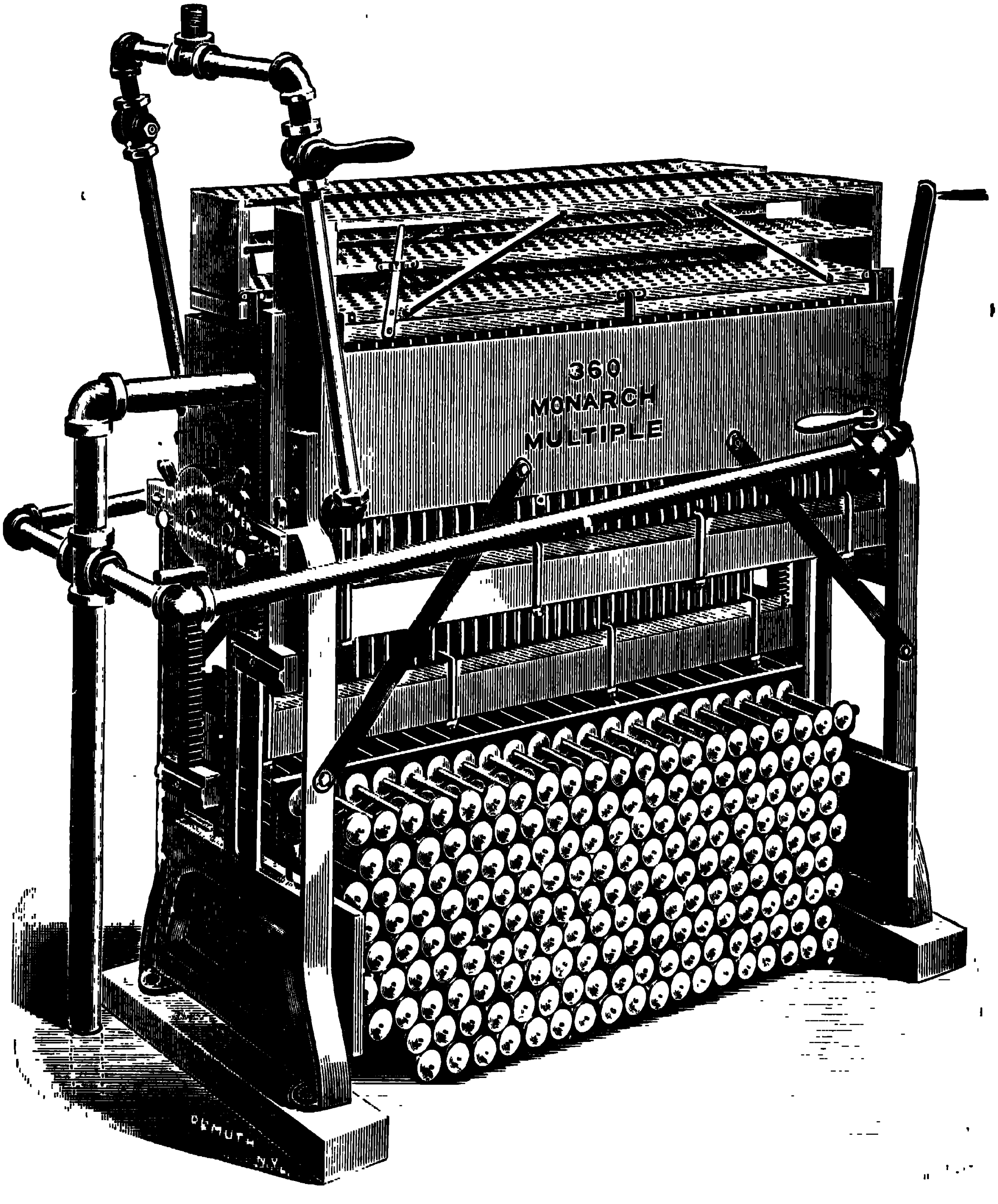

FIG. 188 —360-mould Multiple Candle-machine.

When all the moulds of a machine are to be removed at one time, those at the four corners should be first replaced. In such a case the four corner pistons should be so adjusted as to bring their tips to a perfect level with the tops of the moulds, otherwise the horizontal adjustment of the lower rack may not be retained. The pistons are made of seamless drawn-brass tubing and are soldered to the tips and fitted with a flange at the lower end, by means of which they are secured to the lower rack.

The wicks pass from the spools, or bobbins, through the hollow piston into the moulds, the necessary tension being furnished by the accurate drilling of the holes in the tips. Ample bearing of the tip in the mould is provided. The tip is the conical part of the mould that conforms to the tip or top of the candle.

The strain of raising the pistons falls directly on the lower rack, or platen, which is the common follower on which the ends of the pistons rest.

The upper racks are two wooden contrivances that receive the candles from the moulds. When the lower rack is raised, the pistons force the candles upward out of the moulds into them. The upper racks consist of a sliding-board at the bottom and two or more stationary receiving-boards. The number of the latter varies with the length of the candle a machine is to cast, as many as five being necessary for very long candles. When the candles are raised out of the moulds by the pistons, they pass upward through circular apertures in these racks until the shoulders of the candle tips are slightly above the sliding board. By the aid of the cam-lever (*L*, Fig. 185) the sliding-board is moved horizontally, whereby the shoulder of each candle is brought to rest upon the sliding-board's aperture and locked in this position. Since the receiving-boards are stationary, the horizontal movement of the sliding board beneath them tilts the candles slightly. The apertures of the receiving- and sliding-boards being then no longer in vertical lines, the candles cannot drop down through the apertures of the latter. Thus the entire casting is suspended over the moulds, locked in the upper racks. The upper racks being removable, their bearings simply resting in the tray-slots, the product of the machine is removed by the mere lifting of the two racks. The candles are removed from the racks by inverting them.

The horizontal movement of the sliding-board above described, however, carries the continuous wicks away from the centres of the moulds. But the wicks must be recentred before the next pouring. When the upper racks are moved back in the tray-slots as far as the sliding-board was moved forward the wicks are centred. The slots in the edges of the tray allow the racks to be moved by hand just far enough to bring the wicks back to the centres of the moulds.

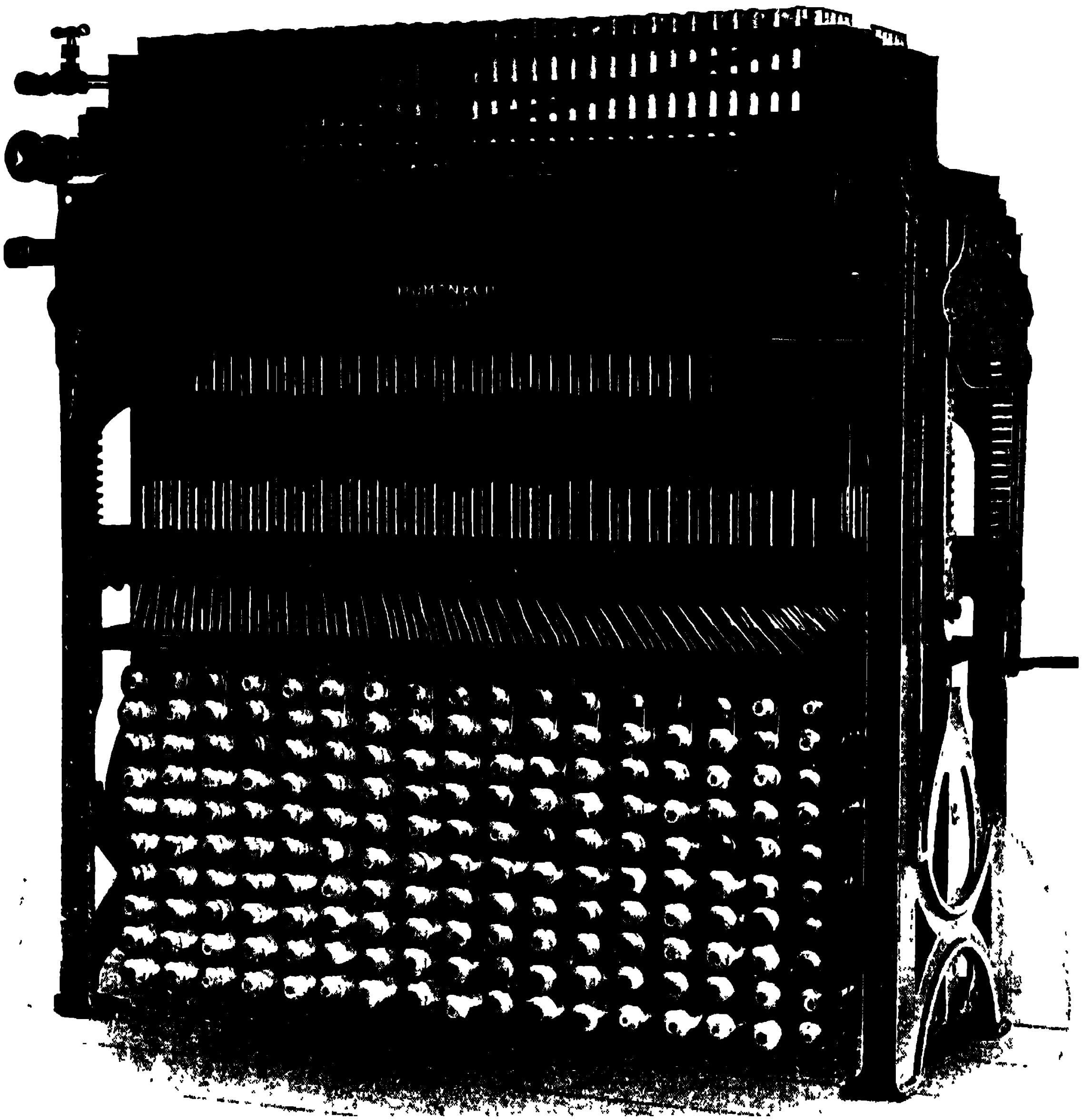

FIG. 189.—A 360-mould Candle-machine.

The following directions for operating the upper racks should be followed: Place them on the machine so that they will rest in the slots in the vertical edges of the tray, being careful that the cam-levers are at the outside, as the racks are *rights* and *lefts*. Push each rack toward the rear end of the machine as far as the slots will permit. Raise the cam levers to a perpendicular position and turn the crank of the machine until the tips of the candles are a little above the sliding-board; then press down the cam-levers as far as they will go and let down the lower rack of the machine to the bottom, when the candles will be left fast in the upper racks. Then pull the upper racks toward the front of the machine as far as the slots will permit, repeating the operation for each casting.

The tray comprises two horizontal channels over the box or chest containing the moulds. Into the tray the material is poured, with the upper ends of the moulds open.

The wooden spools, or bobbins, serve to hold the supply of wick, which is continuously furnished to the moulds through the tubular pistons. The spools are free to rotate on horizontal pins, so arranged under the lower rack as not to interfere with each other or with the operation of the rest of the moulding-machine.

Setting Up and Wicking Candle-machines.—Referring to Figs. 181 and 182, the machines should be set about four feet apart, centre to centre, according to the size of the machines and space available, the dimensions stated, however, providing ample room for operation. The character of the connections of the double box type are indicated in Fig. 186.

To wick the moulds of the machines, elevate the lower rack, by turning the crank, until the tips of the pistons are on a level with the mould tops. Push the double wicking wire through the hole in the piston-tip until the loop protrudes below the lower rack. Put the end of the wick through the wire loop and extract the wicking wire, which will bring the wick with it. Tie a knot in the end of the wick and pull down the knotted end of the wick into the tip. The first run or casting of candles will be wickless. A piece of thin copper wire about four yards long makes a good wicking wire when doubled so as to make a long loop.

Moulding. The important considerations of candle-making are the temperature of the melted stock and of the moulds, and the rapidity and degree of cooling of the moulded candles. Paraffin in the pure state can be heated to 300° Fahr. (148.80° C.) without affecting its color; but pure stearic acid should not be heated above 200° Fahr. (93.30° C.), as any greater heat is apt to bring about discoloration. The right temperature for the particular stock employed is determinable by experiment. Pure paraffin, as well as paraffin and stearic acid, should be heated to a temperature varying from 165° to 190° Fahr. (73.80° to 87.80° C.), according to the mixture used. The greater the percentage of paraffin the nearer the temperature may be brought towards the minimum given. The water for cooling pure paraffin candles in the moulds should be at a temperature of from 50° to 60° Fahr. (10° to 15.50° C.). To cool composite candles, warmer water may be employed, the degree of warmth depending upon the proportion of paraffin to stearic acid. It may be accepted as a rule that whenever the candles stick to the moulds it is because the water was too warm or it was not applied soon enough after the filling of the moulds.

The moulds of the machine should be heated for pure paraffin wax or composite candles to a temperature approaching their melting-point. The moulds being so heated, turn off the supply of steam, then pour into the tray sufficient stock to fill the moulds and to leave a depth of about half an inch of stock standing in the tray, the extra stock being to prevent the candles from being hollow at the ends by feeding the shrinkage during the process of cooling.

As soon as the required amount of stock has been poured into the machine, open the water-valve, filling the box as soon as possible.

Paraffin and composite materials are prepared at a higher degree of heat than tallow and stearic acid, and should be cooled more quickly in the moulds. The highly crystalline structure of stearic acid renders it liable to crack if chilled too suddenly, or to crystallize if chilled too slowly, whereby the appearance, if not the structure of the candle, is impaired. Slow cooling favors the formation of a crystalline structure, which is a consideration of importance with stearic acid, but of less importance with paraffin, and which in all cases is to be avoided.

When the box is filled, if economy of water is desirable, check down the water-valve, but at least a slight flow of water through the machine should be maintained until the candles are ready to draw. Where water is plentiful, however, as much water as the overflow will carry off should be admitted into the machine continuously.

As soon as the stock in the tray commences to harden, it should be loosened from the ends and sides with a knife or other sharp instrument, otherwise it will stick to the tray. When the stock in the tray has become firm, remove it with a scraper especially made for that purpose, leaving the tray perfectly clean and the ends of the candles smooth in the moulds. If this is properly done, no butt-sawing machine is necessary.

When the candles are cold enough to be started from the moulds, place the upper racks on the top of the machine, taking care to put each rack where it belongs, as the racks are rights and lefts. The racks being in their proper places, loosen the catches or cam-levers until the apertures in the receiving- and sliding-boards are in a vertical line, and when this is done, the crank of the machine should be turned until the candles are lifted by the pistons clear of the lower or sliding board of the upper racks; then close the catches or cam-levers and drop the crank back to its original position, the run of candles being locked in the upper racks by the closing of the catches and the machine made ready to turn on the steam for heating the moulds for the next operation.

The wicks connecting the candles in the racks and the moulds must not be cut until the candles in the moulds are sufficiently hardened to retain the wicks upright. The wicks being cut with a long, sharp knife, the upper racks are removed and the tray cleaned of the excess stock for the next operation. By reference to Fig. 190 the general construction of English candle machinery and procedure of operation may be seen.

Operation of Machine.—Referring to the candle-machine shown in Fig. 185, the mode of operation, which is common to all the machines illustrated, may be described as follows: The wicks are first passed up through the tubular pistons *C*, thence through the moulds, and finally secured at the upper ends of the latter, after which act the follower *D* is either raised or lowered by turning the crank *G* so as to produce candles of the desired length, the

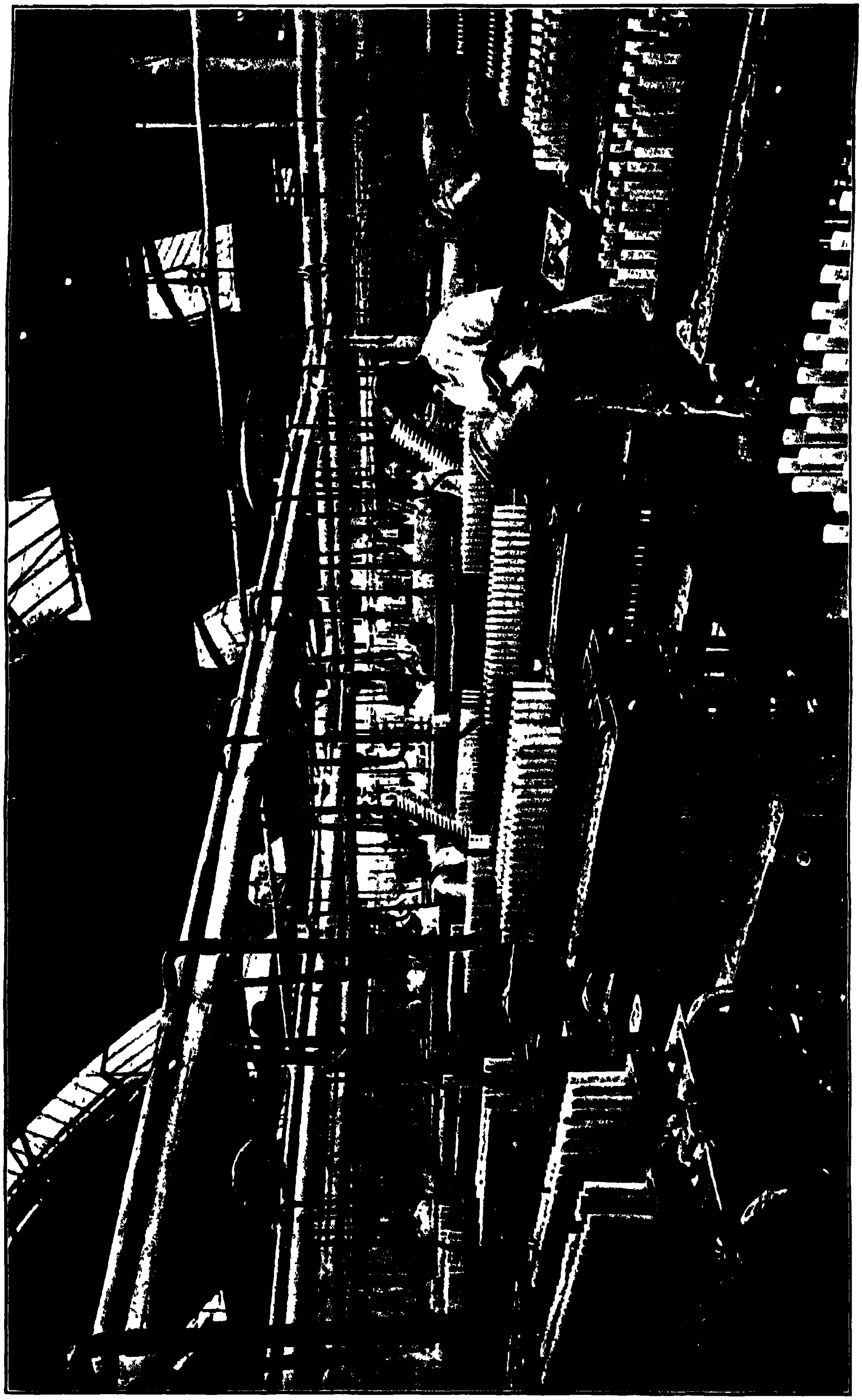

FIG. 19c.—Candle-moulding Room. (Price's Patent Candle Co., Ltd.)

follower being maintained in this position by properly adjusted gauges or stops. The melted stock is now poured into the channels of box *B* at *FF′* and runs directly into the open upper ends of the moulds, the latter being made warm by the admission of steam or hot water to the interior of the box. The stock descends into the moulds until it is arrested by the conical recesses in the heads of the pistons, and when all the moulds are filled the supply of steam or water is shut off and cold water is admitted to the chest for the purpose of cooling the candles, which operation usually takes about twenty minutes. The candles having been thus cooled and hardened, the surplus stock is speedily cut away from the channels, so as to leave each candle at liberty to be raised out of the mould, but previous to doing so, the racks *J* and *K* are placed in position upon the chest *B*. These racks are furnished with horizontal boards having circular holes in them, which holes are accurately in line with the moulds and are sufficiently large in diameter to admit the candles freely. These precautions having been taken, the operator turns the crank *G* in such a manner as to elevate the follower *D* and the pistons attached thereto, the result being to force the candles out of the moulds and drive them into the racks *J* and *K*, as seen in the illustration. Each rack has a slide which is shifted longitudinally a slight distance by a cam-lever *L*, so as to lock or clamp the candles therein, the wick extending uninterruptedly from the moulds down to the spools *H*. The moulded candles being thus locked in position, the follower *D* is lowered until it again rests upon the stop, when the above-described operations are repeated. Sufficient room is left at one end of the racks to allow the melted stock being poured into the channels. Before the second run of candles becomes cooled, the wicks of the first run are cut by passing a sharp knife horizontally beneath the racks, and the charged racks *J* and *K* are removed from the box *B*, emptied of the candles, and then returned to receive the second run as soon as they are hardened and elevated in the manner just described. During the cooling process the water admitted into the chest *B* finds its way out through numerous orifices that open into the overflow troughs *M*, which are fitted with waste pipes for carrying it away.

Candle-makers' Tools.—Candle-makers' requisites include the swimmer for dipping the material from the kettle; the pouring-bucket for filling the moulding-machine; the steel scraper for cleaning the tray of the excess material, which, however, does not trim the ends of the candles; the steel cutter, which simultaneously removes the excess material and trims the ends of the candles; the steel knife for cutting the wicks; and a standard thermometer. The cutter has come into quite general use, on account of its utility for the double purpose mentioned. Most candles can be thus trimmed in the moulds, which does away with the need of a butt-sawing machine. It is made to fit the width of the tray-channels, so that the

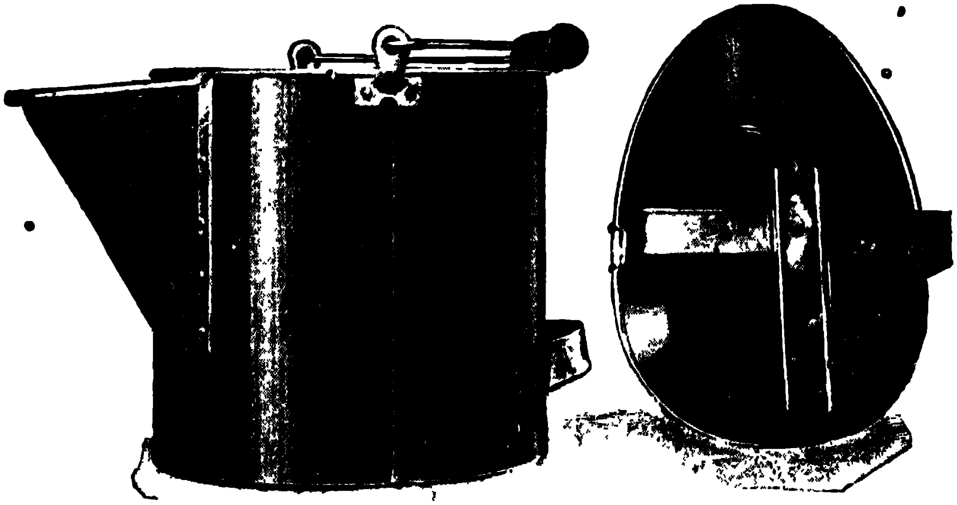
FIG. 191.—Pouring-bucket and Swimmer.

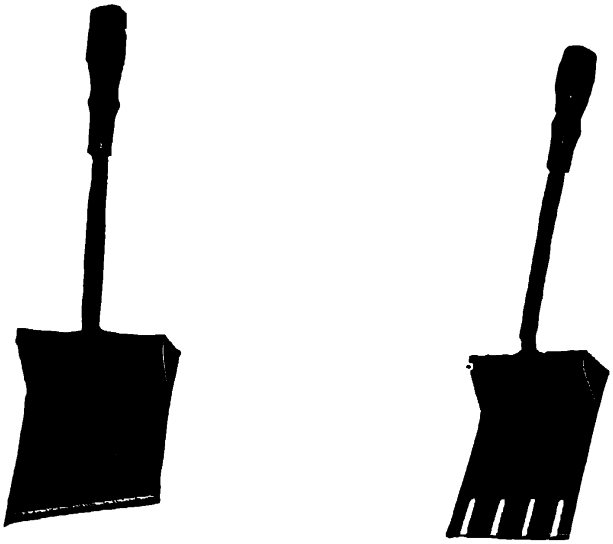
FIG. 192.—Steel Cutter and Scraper.

excess material can be quickly cut away. Pure stearic-acid candles are too brittle to permit of the cutter's use; so with this material

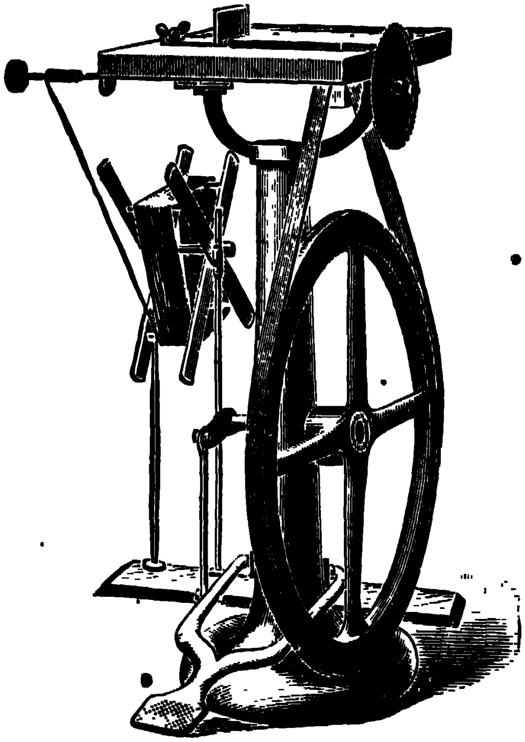

FIG. 193.—Combined Butt-saw and Wick-winder.

the scraper is employed to remove the excess stock and the butt-saw (Fig. 193) to trim them after they are moulded. Candle-butt sawing machines are not now so much used as formerly, because most of the candles can be trimmed in the moulds as just described. This elimination is in itself a decided step toward simplicity and economy of manufacture.

Sizes of Candle-machines.—Candle-machines are rated according to the number and size of candles produced at a single casting. A combination machine will cast candles differing in diameter and length and is especially constructed to mould a varied product. By the use of gauges the pistons can be adjusted higher in the moulds, permitting the casting of shorter candles than those corresponding to the full length of the moulds. But when there is much taper to the mould, or the material becomes very thin from heating, as is the case with pure paraffin, the material will escape by the piston if thus adjusted.

Manufacturers of experience provide a machine for every size of candle they manufacture, having learned that in the end it is the most economical plan. There has been great improvement in construction between the present and the older machines, which were of less capacity. The development was to a great extent brought about by the demand for machines of greater capacity and strength, together with the introduction of new materials for candle-making. Tallow, for instance, is easy upon a machine, while on the other hand, paraffin is severe, and where a machine of 26 moulds was formerly deemed of sufficient capacity, the demand of to-day is for a 240-mould machine for ordinary sizes and for a 360-mould for smaller sizes. The 240-mould machine seems to be about the natural limit of a machine for casting candles in most general use, viz., from fourteen to four to the pound, and the 360-mould machine for smaller candles, such as Christmas or toy candles not over 7 inches long and $\frac{5}{8}$ of an inch in diameter.

Inasmuch as the highest-capacity machines can be operated at the same cost for labor as those of smaller capacity, it is the most economical plan to equip a plant with large machines whenever conditions peculiar to each establishment and volume of output warrant their use. 144- and 240 mould machines are commonly used for casting short, thick candles about 7 inches long and varying in diameter from $\frac{5}{8}$ to $1\frac{1}{4}$ inches, commonly styled coach and carriage candles. Special machines are made for casting large sizes (see Figs. 179 and 194) weighing from three to a pound to three pounds each or more. Such candles have heretofore been manufactured by the laborious hand process, which still obtains in many countries. As a general rule upper racks are not used with machines casting candles above one pound in weight. It would hardly be practicable for operators to handle upper racks full of candles of a larger size. Such machines, therefore, are equipped with short brass pistons, which raise the candles about 7 inches out of the moulds, enabling the operator to remove them by hand with facility. Being minus upper racks, some device must be substituted for holding and centering the wicks. The wicks in such machines are held in place by pins that pierce the edges of the tray opposite the centre of each row of moulds. Furthermore, the absence of upper racks and the use of short pistons enable the height of such machines to be reduced, thus facilitating their operation by reason of the attendant compactness. The pistons may be elevated by rack and pinion as in Fig. 194 or by lever as in Fig. 179.

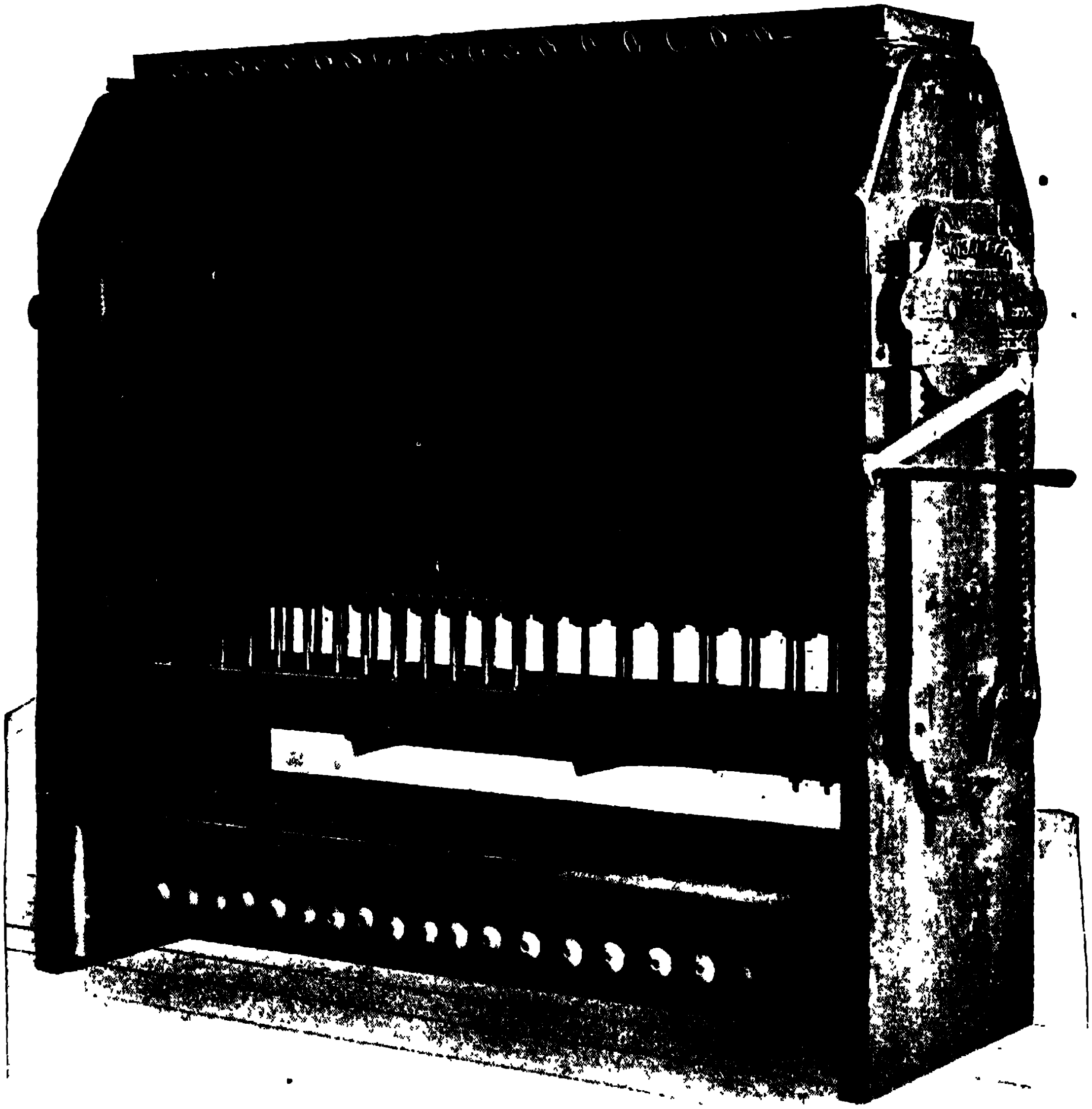

FIG. 194.—36-mould Machine for One-, Two-, and Three-pound Candles of any Kind of Material.

Bleaching and Polishing Candles.—For ordinary candles the smoothness and polish imparted to the interior of the tin or tin-alloy moulds of the candle-machine suffice to impart to the candles a high degree of lustre and smoothness Exposure to sunlight for varying periods is the common method employed to remove any yellowish tinge. The polishing-machine consists essentially of two grooved wheels mounted on a horizontal shaft before which is located a feed box into which the candles to be polished are placed. The grooved wheels carry the candles one by one from the feed-box past a revolving saw which removes the butts and deposits them of uniform length upon a bedplate between the rods of an endless frame with linked sides, kept in motion by cog-wheels. Over this bedplate they roll under a revolving buffer moving at right angles to the endless belt carrying the candles, which gives them a vigorous brushing from end to end and imparts to them a beautiful porcelain finish as they pass toward the end where they roll off into the packer's box. The best grades of candles, as a rule, are stamped with the name of the maker, and in some instances the trade name of the candle, "Composite," etc.

Classification of Candles.—Candles are classified by size according to the number required to weigh a pound, which is subject to great variation according to the dimensions of any given size. In Table XXXI, given on the opposite page, may be seen the relation existing between candles of given dimensions and the corresponding number required to weigh one pound.

Candles are moulded in a great variety of shapes, the most common shape being that of a plain cylinder of varying diameter and length. A great variety of ornamental forms for religious and decorative purposes are moulded in numerous prismatic cross-sections, as square, triangular, octagonal, etc. They may also be moulded longitudinally or spirally fluted. The latter form was originally turned in a lathe, but is now made in moulds and in ejecting the candles from such moulds the candles are made to rotate. Decorated candles are of every color, shape, and design. Red, green, pink, white, and yellow are the most popular colors. The decorations are made of wax and put upon the candles by hand. The wax is colored, so as to give the decorator's artistic taste the widest possible range. Thus there will be red holly-berries and green leaves twining around the white Christmas candle. There will be white designs, red background, and so on indefinitely. The structure of the candle may be modified by longitudinal perforations or channels extending the entire length to permit the descent of melted matter

TABLE XXXI. CANDLE DIMENSIONS—(*English.*)

Weight.	Length.	Diameter.
4 lbs. each	46 inches	2 inches
3 “ “	40 “	1¾ “
2 “ “	39½ “	1½ “

Number to Pound.	Length in Inches.	Diameter in Inches.
Long 1	36	1
Short 1	20	1⅜
Long 2	16	1 1/16
Short 2	12	1¼
Long 3	15	15/16
Short 3	12	1 1/16
Long 4	13	⅞
Short 4	9	1
Long 5	12	¾
Short 5	10	⅞
Long 6	12	¾
Short 6	7¼	15/16
Long 8	12	⅝
Short 8	5½	15/16
“ 10	6	13/16
Long 12	12	½
Short 12	6	¾
Long 16	6⅞	⅝
Short 16	4¼	¾
“ 18	6⅜	⅝
“ 3	7	5/16
Short 5	5	1 3/16
Long 6	6	1
Short 6	3¼	1 5/16
“ 10	4	15/16
“ 8	4	1 1/16
“ 16	4	¾
“ 32	4	½
“ 48	4	7/16
“ 72	4	⅜
“ 90	3	⅜
“ 120	2½	⅜
“ 200	2½	¼

in the interior and not to “gutter” on the outside. This necessitates the use of material of high melting-point and is adapted only to candles of larger diameter than the average. The channelling is effected by attaching shafts to the piston-head corresponding in number and location to the channels in the candle. The yield of candles from a given weight of stock may be considerably increased by crutching the melted stock previous to casting, whereby air is

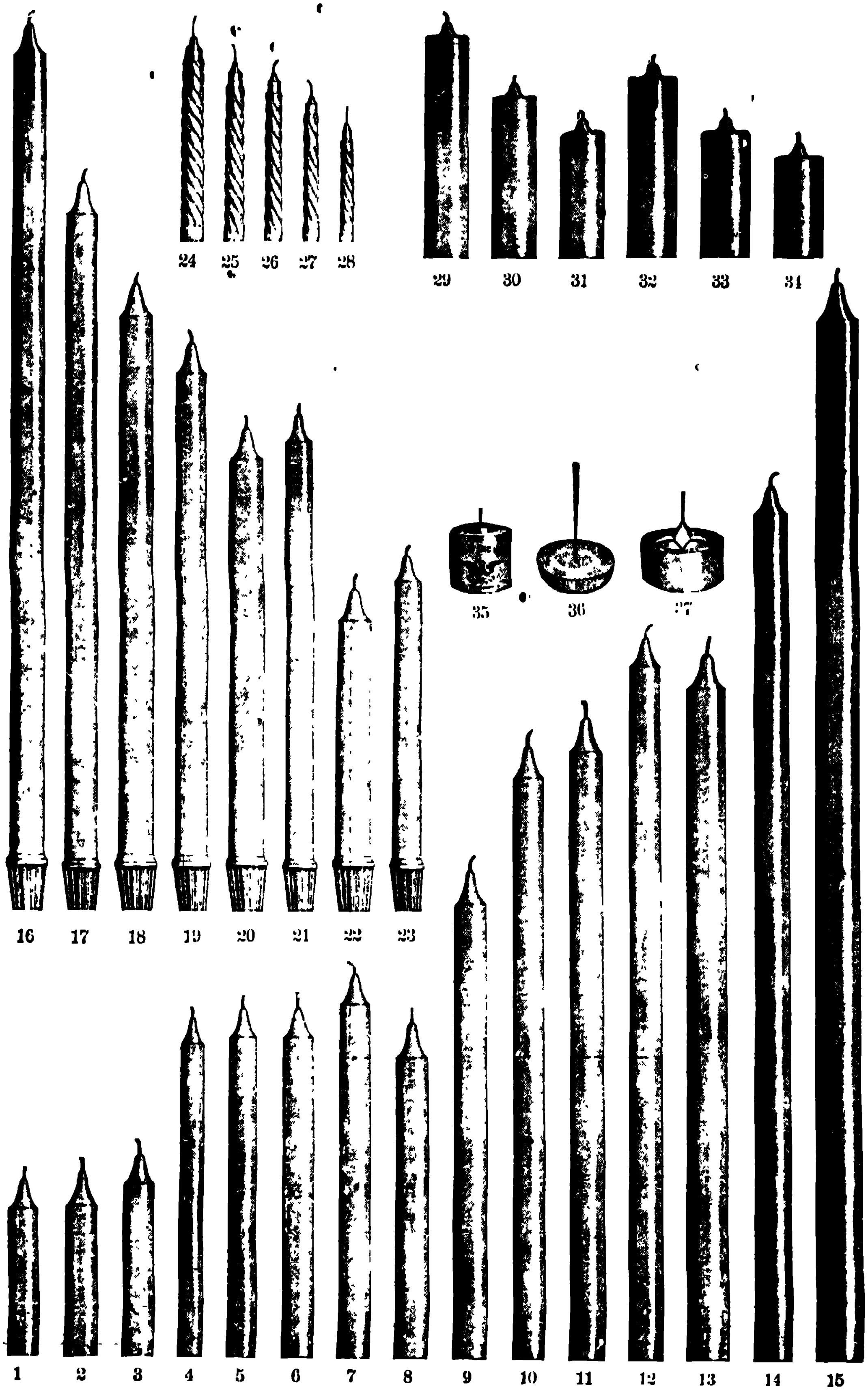

FIG. 195.—Stearic Acid or "Stearin" Candles. Representative Shapes and Sizes.

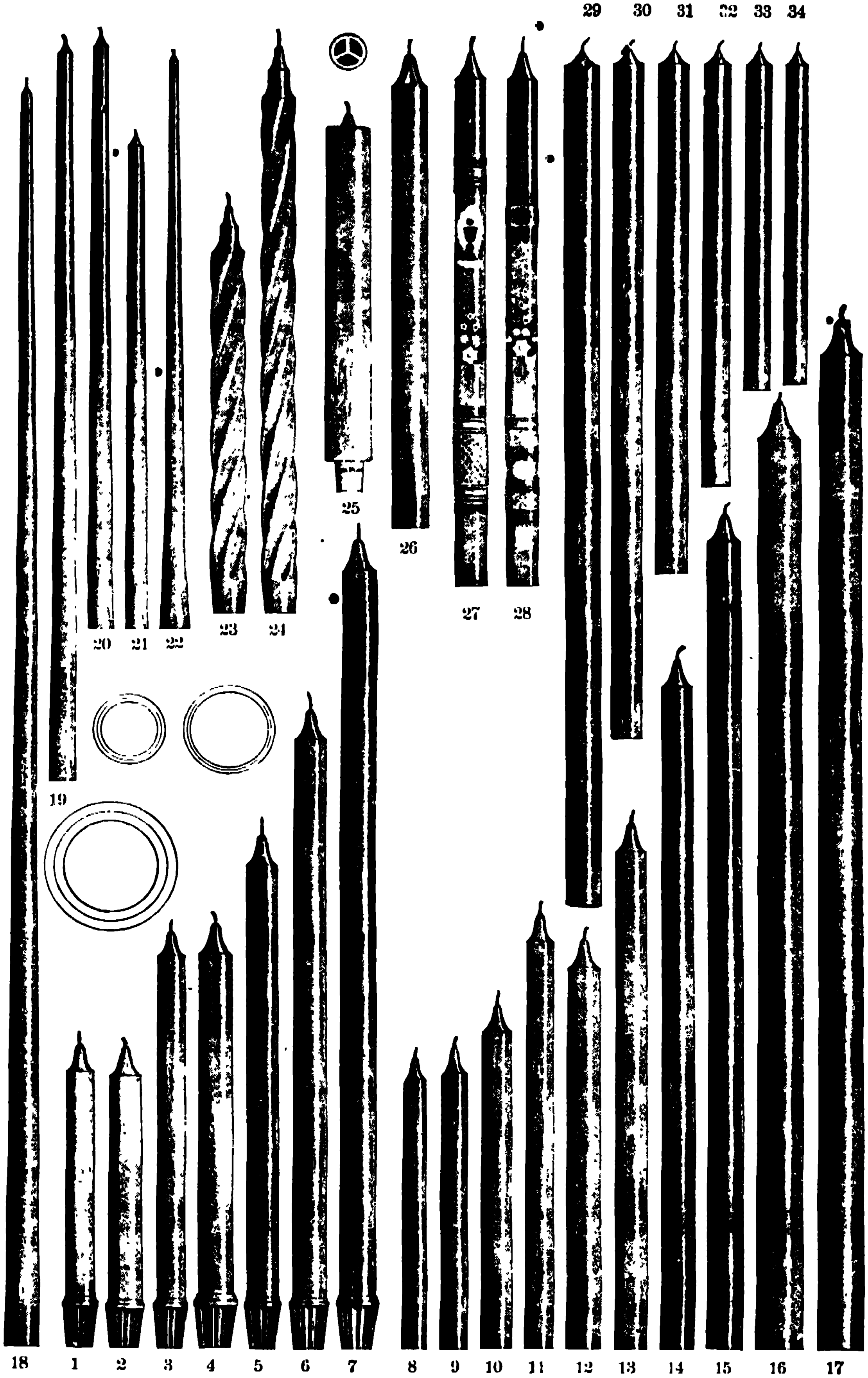

FIG. 196 —Moulded "Beeswax" Candles Representative Shapes and Sizes.

incorporated and the volume occupied by a given weight of stock is increased. The method of preparing the stock prior to moulding is analogous to that of floating soap. Such candles are consumed very rapidly.

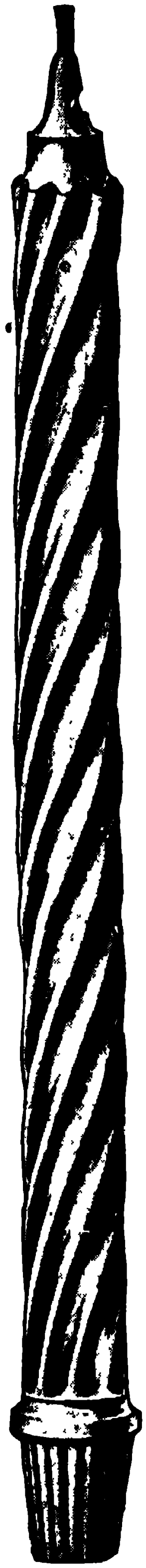

FIG. 197.—Renaissance Self-fitting-end Candle.

Long tapering candles are made by dipping and rolling upon a marble slab. The base of the moulded candle in its finished form is of slightly greater diameter than the top; as they are moulded bottom end up, this provision admits of their easier ejection from the mould. In all cases by the length of the candle is meant the distance from the base to the shoulder. In Figs. 195 and 196 are shown the more common grades, forms and sizes of candles made by American manufacturers. The following tables, viz., XXXII and XXXIII, relating respectively to stearic-paraffin and compounded beeswax candles, are explanatory of Figs. 195 and 196.

Self-fitting Ends. — Candles may be made with tapering butts to enable them to fit any holder either by use of the tapering-machine shown in Fig. 201 or by the use of additional moulds inserted in the fluted or regular candle-mould. A type of candle of this description is shown in Fig. 197. The first self fitting-end candle is attributed to Field, who in 1861 patented his device for giving candles a conical end or butt by moulding. His device has been greatly improved upon until at the present time the most satisfactory methods for making self-fitting ends is by use of individual moulds inserted in the tops of the cylindrical moulds. The use of these moulds is shown in Fig. 198. It has become a custom to have fancy candles made with self-fitting ends, but the self-fitting end is applicable to any kind or style.

A machine for casting candles with self-fitting ends differs in construction in certain respects from a machine for casting plain-end candles. By a self-fitting-end machine is meant one which casts candles with ribbed and tapered ends that adjust themselves to any candlestick in ordinary use. The extreme end of the self-fitting portion of the candle is the least diameter of the candle, excepting the diameter at the tip. It therefore becomes necessary to remove the self-fitting-end part of the mould before the candles can be elevated into the upper racks. As the distance between the bottom of the tray and the bottom of the upper racks in the regular

TABLE XXXII.—STEARIC ACID OR "STEARINE" CANDLES. DIMENSIONS OF REPRESENTATIVE SHAPES AND SIZES.

Number in Fig. 105	General Description.	Number to Pound *	Length in Inches.	Diameter in Inches.
1	12 oz.	12	4½	13/16
2	Short shape.	12	4¾	7/8
3	Long "	12	5¼	13/16
4	Tube.	12	8¼	5/8
5	Long shape.	8	9	3/4
6	14 oz.	6	9	13/16
7	Long shape.	6	10	7/8
8	Short "	6	8¼	7/8
9	" "	1	12¼	7/8
10	Long "	4	16	13/16
11	Short "	3	15¼	15/16
12	Long "	3	19	7/8
13	Short "	2	18½	1⅛
14	Long "	2	23	15/16
15	Short "	1	28	1 3/32
16	Self-fitting - Long shape.	2	23¼	15/16
17	" " "	3	19	7/8
18	" Short "	3	16¼	15/16
19	" Long "	4	15	7/8
20	" Short "	1	12½	15/16
21	" Long "	6	13	3/4
22	" Short "	6	8½	15/16
23	" Long "	8	9¼	3/4
24	Table-Toy. Assorted colors	18 No. per Box	5½	5/8
25	" " "	24	4¼	1/2
26	" " " "	36	4½	7/16
27	" " " "	48	3⅞	7/16
28	" " " " . . .	72	3⅛	3/8
29	Coach.	5	6¼	1⅛
30	"	7	4⅝	1⅛
31	"	9	3⅝	1⅛
32	"	5	5¼	1¼
33	"	7	3¾	1¼
34	"	9	3	1¼
35	Night-lights. Burns 8–10 hours.			
36	Starlight No. 1 " 4 "			
37	" No. 2 " 10 "			

* 10 oz. per lb.

TABLE XXXIII.—MOULDED "BEESWAX" CANDLES: DIMENSIONS OF REPRESENTATIVE SHAPES AND SIZES.

Moulded "Beeswax" Candles. Self-fitting Ends.

Number in Fig 196.	General Description.	Number to Candle Pound.*	Length in Inches.	Diameter in Inches.
1	....	8	8¼	¾
2	Short shape..................	6	8¼	⅞
3	Long "	6	11⅜	¾
4	Short "	4	11½	⅞
5	Long "	4	14	13/16
6	" "	3	17½	⅞
7	" "	2	23	15/16

* 14 oz. per lb.

Moulded "Beeswax" Candles. Plain Ends.

Number in Fig 196.	General Description.	Number to Candle Pound.	Length in Inches.	Diameter in Inches.
8	Tube......	12	8	⅝
9	Long shape.....	8	8¼	¾
10	Short "	6	9½	13/16
11	Long "	6	12	¾
12	Short "	4	11¼	⅞
13	Long "	4	14⅜	⅞
14	" "	3	18⅞	⅞
15	" "	2	22¾	15/16
16	Short "	1	26	1¼
17	Long "	1	28	1⅛

Tapering Wax Candles. Hollow or Solid at Base.

Number in Fig 196.	General Description.	Number to Candle Pound.	Length in Inches.	A Shoulder.	Base.
18	....	2	34¼	5/16	⅞
19	....	6	20	⅜	¾
20	....	8	16	⅜	11/16
21	....	12	13¼	⅜	⅝
22	....	16	15½	3/16	¾

Renaissance Candles. Made in Beeswax or "Stearine."

Number in Fig 196.	General Description.	Number to Candle Pound.	Length in Inches.	Diameter in Inches.
23	....	6	11	15/16
24	. . ..	4	15½	15/16
25	Top candles..................	3	10¼	1¼
26	Aerated......................	4	12⅞	1
27, 28	Ornamented wax, made in all sizes.			

Beeswax Candles. Hand-Made, Patent Finish.

Number in Fig 196.	General Description.	Number to Candle Pound.	Length in Inches.	Diameter in Inches.
29	Long shape..................	2	22	⅞
30	" "	3	18¾	13/16
31	" "	4	14¼	13/16
32	" "	6	12	¾
33	" "	8	9¼	11/16
34	....	12	9	⅝

TABLE XXXIII.—MOULDED "BEESWAX" CANDLES—*Continued.*

EASTER OR PASCHAL CANDLES.

Weight of Each Candle in Pounds.	Diameter in Inches.	Length in Inches.
2	$1\frac{1}{2}$	32
3	$1\frac{3}{4}$	36
4	$1\frac{15}{16}$	38
5	$2\frac{1}{16}$	42
6	$2\frac{3}{16}$	44
8	$2\frac{3}{8}$	50
10	$2\frac{1}{2}$	56
12	$2\frac{1}{2}$	63
15	3	64
20	$3\frac{1}{2}$	59

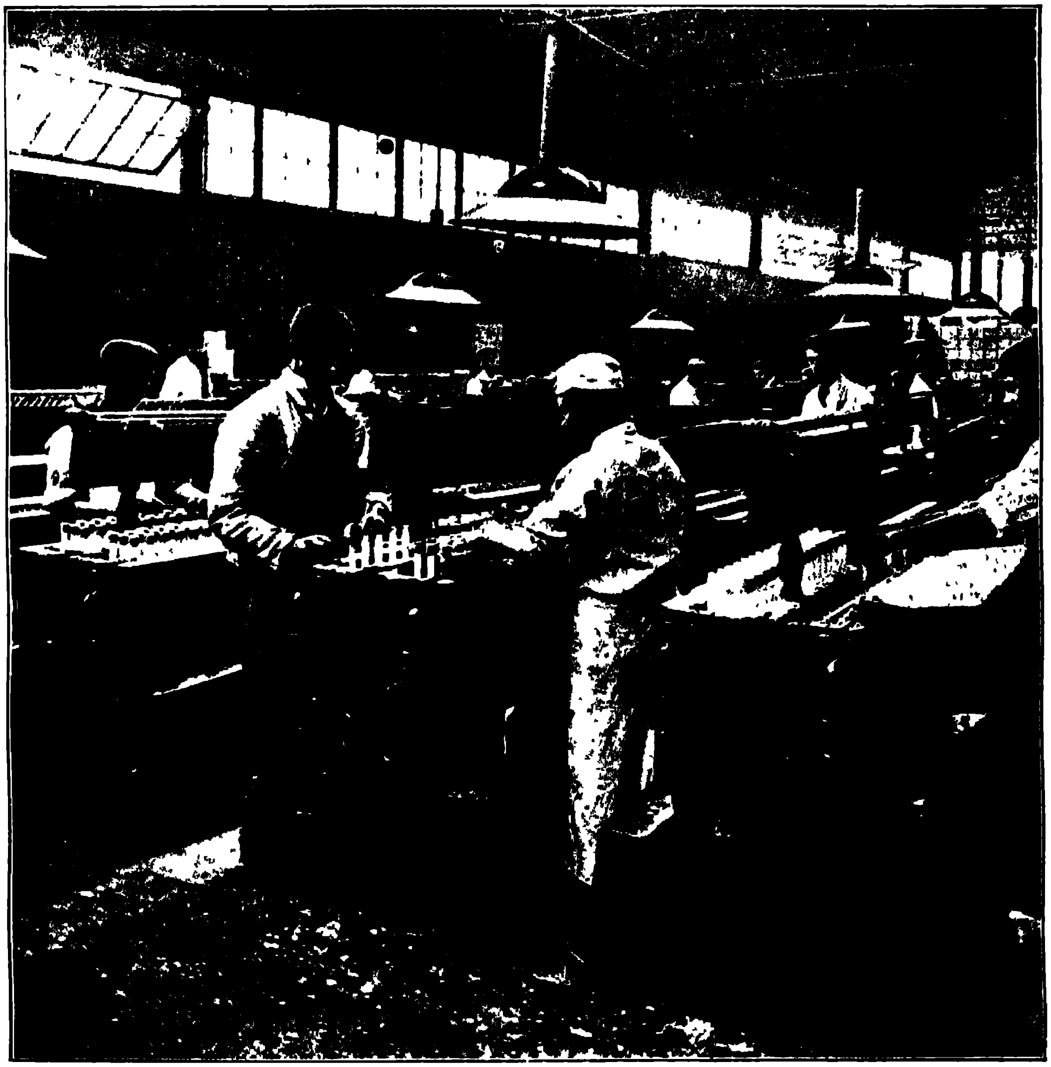

FIG. 198.—Moulding Self-fitting-end Candles. (Price's Patent Candle Co., Ltd.)

machine is not sufficient to permit of this removal, in the self-fitting-end machine the upper racks must be elevated to give room for the

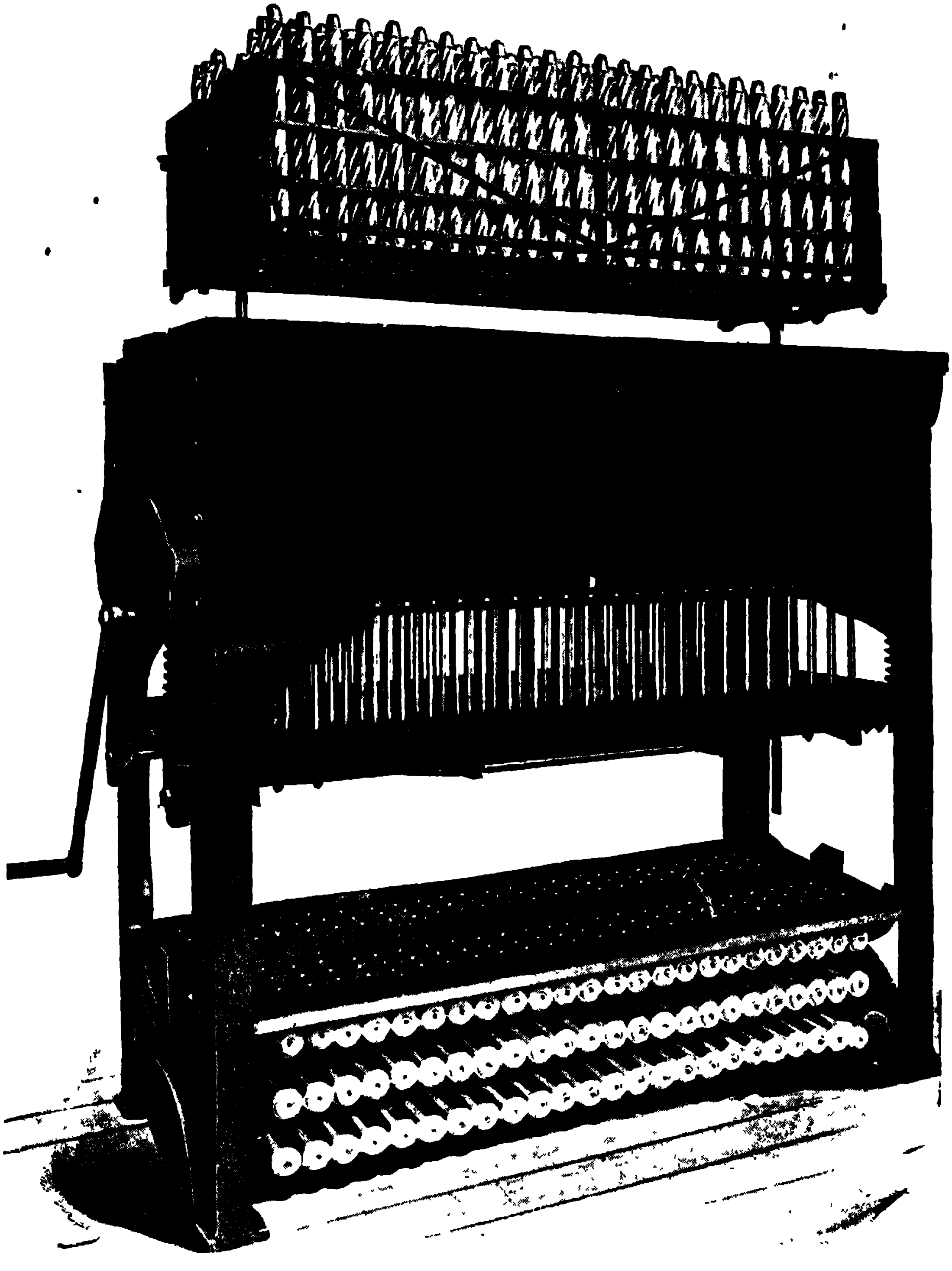

FIG. 199.—Self-fitting-end Candle-moulding Machine.

insertion of the operator's hand when removing and replacing this part of the moulds. The illustrations in Figs. 199 and 200 show the upper racks so elevated. A substantial device is employed for this purpose, which elevates and lowers the lower and upper racks simultaneously. As the tension of the wick is in the tip of mould, the operation of raising the racks does not interfere with its tautness, the play or looseness of the wick occurring between the bobbins and the tip of the mould. The removal and readjustment of the self-fitting-end portions of the moulds are done quite handily, occupying very little time.

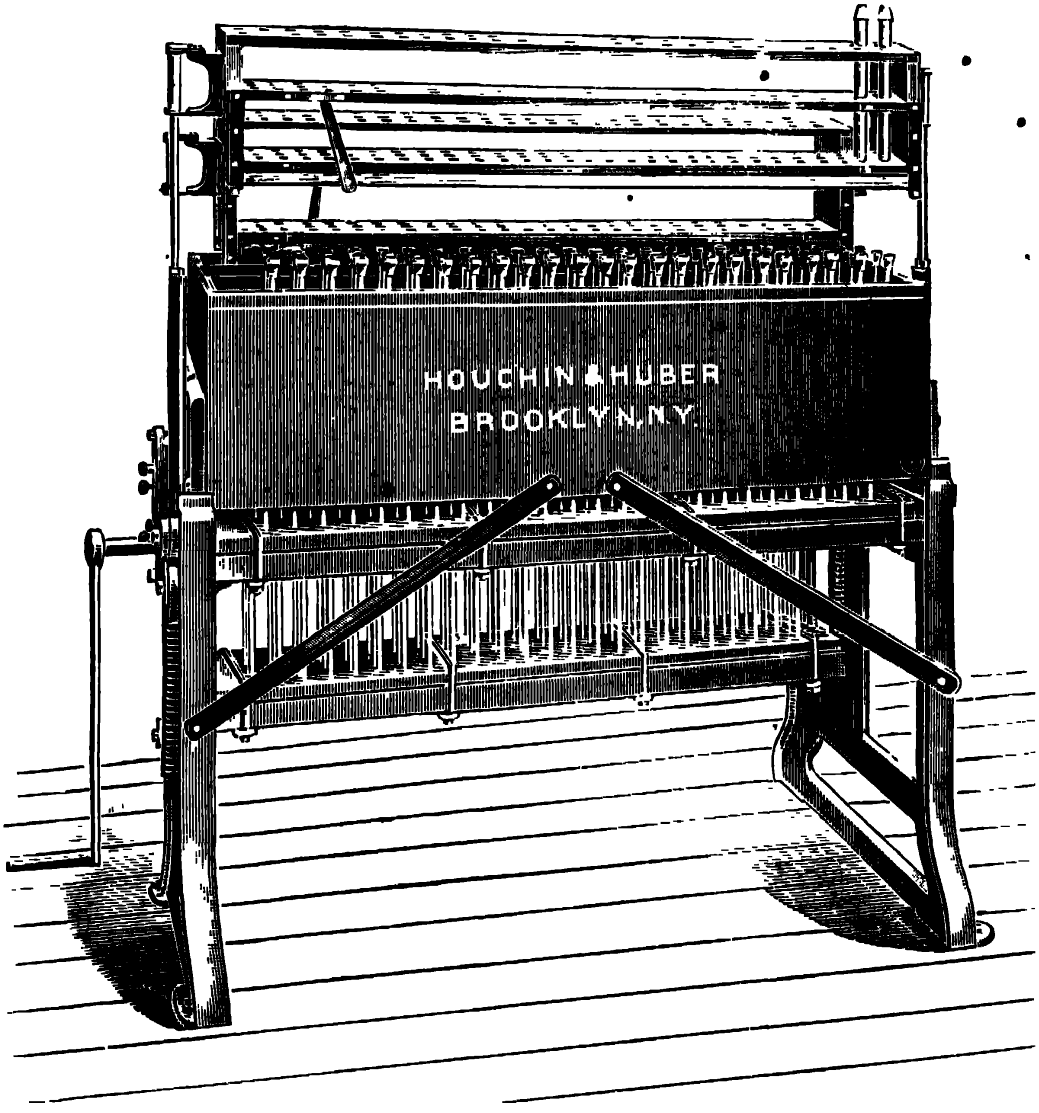

FIG. 200.—144-mould Self-fitting End Candle-machine.

Operation of Self-fitting-end Machine.—After the machine is in position and the wicks adjusted, insert the self-fitting-end caps in the tops of the moulds and fill the machine with material. When the candles have cooled and the excess stock has been removed, turn the crank just enough to raise the self-fitting-end caps from the moulds. Remove these caps. Place the upper racks in position. Raise the candles into them and lock them in place. Let down the lower rack as far as it will go. Pull forward the flat-iron slide on the top of the lower rack until it engages with the perpendicular rods that raise the upper racks. Turn the crank so as to raise the upper racks sufficiently to give the operator's hand room to insert the self-fitting-end caps. When the self-fitting-end caps are in place, let down the upper racks and pull down the wicks, so as to render them taut in the moulds. The machine will then be ready for the second filling. Never raise the pistons in the moulds while the self-fitting-end caps are in position, otherwise the tips will be ruined by bringing them in contact with the sharp inner edges of the caps.

Dipping.— The manufacture of candles by dipping preceded the industrial use of the candle-moulding machine, and the procedure still persists in the manufacture of candles from material which from its character unfits it for moulding. Formerly wax candles for religious purposes, composed solely of beeswax. were made by dipping, the procedure and apparatus for which are illustrated in Fig. 202. The apparatus consists of a vat containing the melted stock, which is surmounted by a frame carrying two pairs of wheels from which is hung, by means of chains and balance weights, a rack for supporting rods, upon which are hung the wicks, which are first saturated with the melted stock and cooled and subsequently dipped and cooled until candles of the desired weight are obtained. Successive stages of cooling and dipping are illustrated in Fig. 202. The weight of a given number of candles is determined by regulating the weights used as a counterpoise.

In dipping candles made entirely of beeswax, variation in diameter of the candle, due to rapid cooling of the wax, is corrected either by rolling the candle, softened by dipping in hot water, upon a smooth surface by means of a flat board or by use of shapers shown in Fig. 203. These are constructed on the principle of scissors and are provided with a sharp cutting-edge. Large candles longitudinally fluted are made by surrounding the middle candle with dummies which are firmly secured to each other by incipient melting induced by immersion in warm water, after which they are shaped. The longitudinal flute may be transformed into a spiral flute by simply twisting uniformly through its entire length the candle made and shaped as described. By mixing beeswax with varying proportions of paraffin or stearic acid, or both, it may be moulded like the ordinary candle.

Unit of Light.—The standard candle of Great Britain (also legalized in the United States) is the practical unit of illuminating power. It shall weigh one-sixth of a pound and burn 120 grains of spermaceti per hour. The standard candle of Germany is otherwise defined: It shall have a diameter of 20 mm., be composed of paraffin, and produce a flame 50 mm. in height. The French standard of light is the Carcel, being the light produced from a lamp of that name:

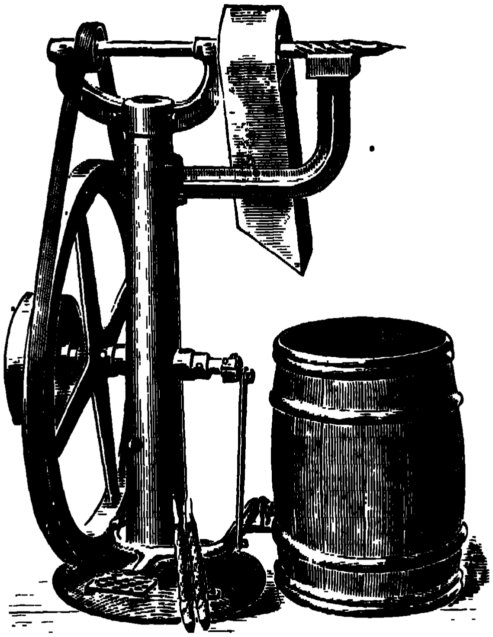

FIG. 201.—Butt-tapering Machine.

1 Carcel = 9.5 standard candles (English).
1 Carcel = 7.5 " " (German).

In the manufacture of the English standard sperm candle it is prescribed * that the wicks shall be made of three strands of cotton plaited together, each strand consisting of 18 threads. The strands

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* By the Metropolitan Gas Referees, London.

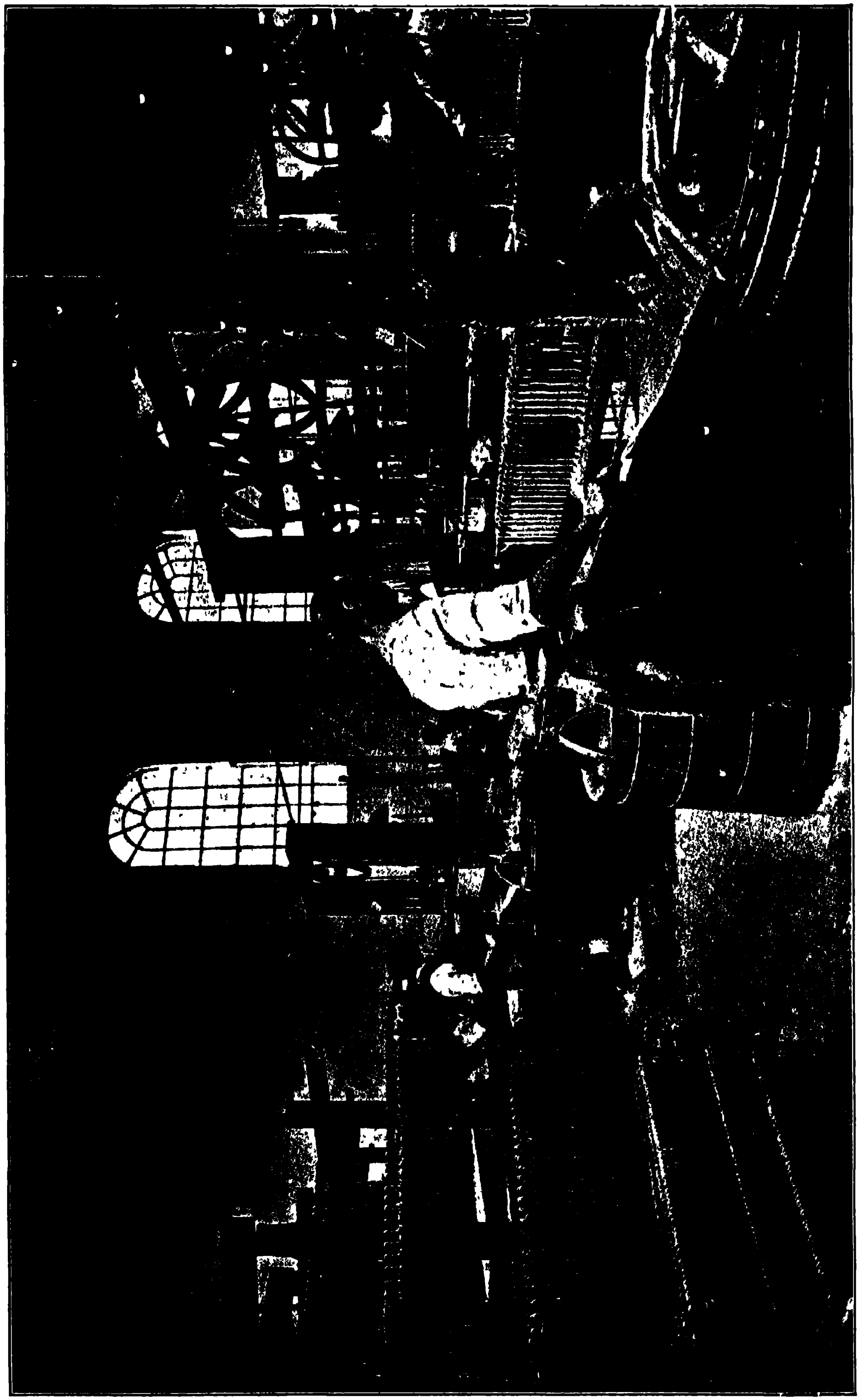


FIG. 202.—Dip-making.

shall be plaited with such closeness that, when the wick is laid upon a rule and extended by a pull just sufficient to straighten it, the number of plaits in 4 inches shall not exceed 34 nor fall short of 32. Each wick shall be of suitable length and looped ready for fixing in the mould. After having been bleached in the usual manner and thoroughly washed, the wicks shall be steeped in a liquid made by dissolving 1 ounce of crystallized boracic acid in a gallon of distilled water and adding 2 ounces of liquid ammonia. They are then to be gently wrung or pressed till most of the liquid has been removed and dried at a moderate heat. Twelve inches of a wick thus made and treated shall weigh not more than 6.5 nor less than 6 grains. The weight of the ash remaining after the burning of 10 wicks which have not been steeped in boracic acid, or from which the boracic acid has been washed out, shall be not more than 0.025 grain.

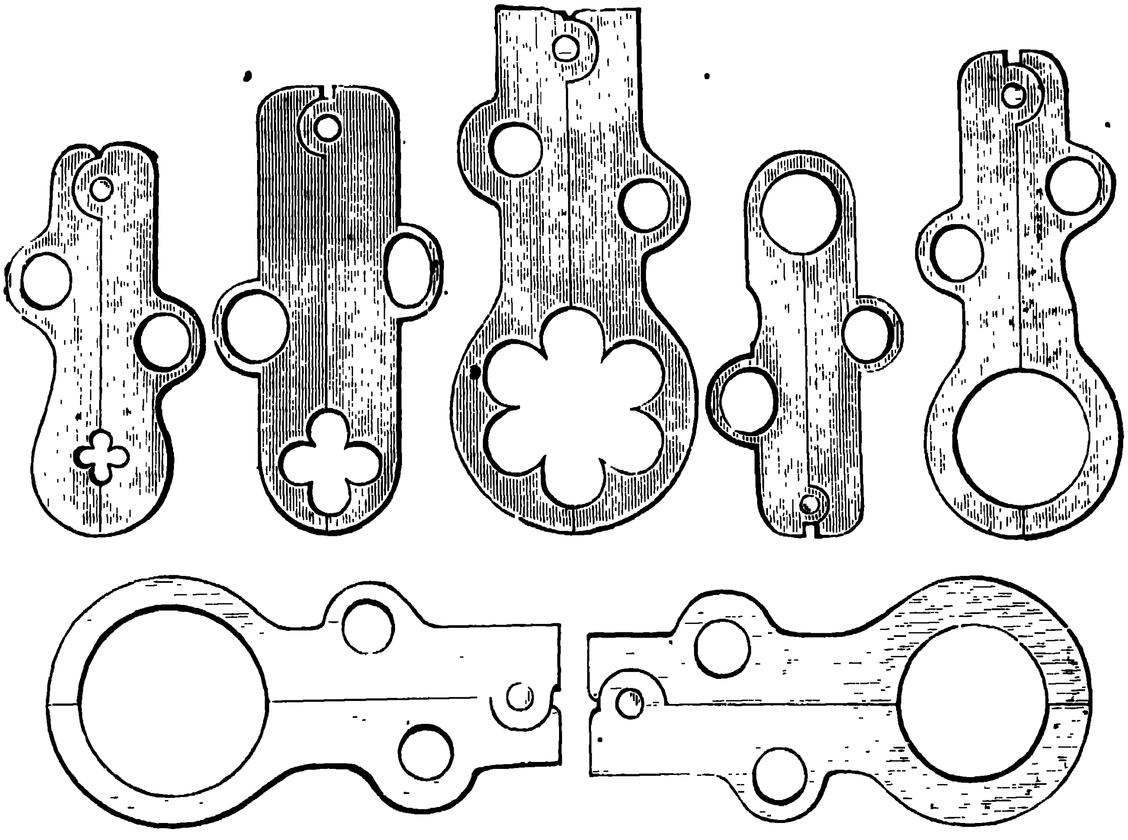


FIG. 203.—Candle-shapers.

The spermaceti of which the candles are made shall be genuine spermaceti, extracted from crude sperm oil, the product of the sperm whale (*Physeter macrocephalus*). it shall be so refined as to have a melting point lying between 112° and 115° Fahr.

Since candles made with spermaceti alone are brittle, and the cup which they form in burning has an uneven edge, it is necessary to add a small proportion of beeswax or paraffin to remedy these defects. It is therefore prescribed that the best air-bleached beeswax, melting at or about 144° Fahr., and no other material, shall be used for this purpose, and that the proportion of beeswax to spermaceti shall be not less than 3 per cent nor more than 4½ per cent.

The candles made with the materials above prescribed shall each weigh, as nearly as may be, one-sixth of a pound, and will be found to answer to the following test: Immerse a candle, taper end downward, in water of 60° Fahr. with a brass weight of 40 grains attached to the wick by a small piece of thread. When a further weight of 2 grains is laid on the butt end of the candle, it will still float; but with a weight of 4 grains it will sink. As the rate of burning of a candle is affected by the force with which the wick is pulled when it is set in the mould, the strain commonly applied by an experienced maker of candles has been measured and is found to be about 24 ounces.

Method of Determining the Melting-point of the Spermaceti.—As the various methods which are used by different refiners of spermaceti for determining the melting-point lead to different results, it must be noted that the temperatures here given as the limits within which the melting point of a sample of refined spermaceti should fall—viz., 112° to 115° Fahr. have been found by the following method, which is known as the capillary-tube method:

A small portion of the spermaceti is melted by being placed in a short test tube, the lower end of which is then plunged in hot water. A glass tube drawn out at one end into a capillary tube about 1 mm. in diameter is dipped, narrow end downward, into the liquid spermaceti, so that when the tube is withdrawn 2 or 3 mm. of its length are filled with spermaceti, which immediately solidifies. The corresponding part of the exterior of the tube is also coated with spermaceti, which must be removed. The narrow part of the tube is then immersed in a large vessel of water of a temperature not exceeding 100° Fahr. The lower end of the tube which contains the spermaceti should be 3 or 4 inches below the surface and close to the bulb of a thermometer. The upper end of the tube must be above the surface and the interior of the tube must contain no water. The water is then slowly heated, being at the same time briskly stirred, so that the temperature of the whole mass is as uniform as possible. When the plug of spermaceti in the tube melts, it will be forced up the tube by the pressure of the water. The temperature at the moment when this movement is observed is the melting point.

CHAPTER XIV.

GLYCERIN.

Historical. Glycerin Obtainable from Fats and Oils. Economic Position of Waste Lye. Waste Lye. Yield. Purification. Procedure. Concentration under Atmospheric Pressure. Principles of Evaporation. Vacuum Evaporators for Waste Lye. Evaporating-plant. Foster Waste-lye Evaporator. Operation. Jobbins Waste-lye Evaporator. Construction. Concentrated Waste Lye. Operation. Products of Concentration. Utilization of Recovered Salt. Crude Glycerin. Distillation of Crude Glycerin. Construction of Reheater and Still. Operation of Still. Expanded and Reheated Steam. Products of Distillation. Construction of Concentrator. Concentration and Utilization of Sweet-water. Concentration of Refined Glycerin. Clarification of Refined Glycerin. Utilization of Glycerin Foots. Garrigues Process of Recovering Glycerin. Operation of Crude Glycerin Plant, Garrigues System. Garrigues Double-effect Evaporator. Garrigues' Process of Glycerin Distillation. Apparatus. Distillation of Crude Glycerin, Garrigues System. Joslin Apparatus for Distillation of Crude Glycerin. Operation. Yield of Glycerin. Glycerin: Origin; Composition; Properties. Nitro-glycerin. Uses of Glycerin.

Historical.—Scheele in 1779, when preparing lead plaster by heating olive-oil with litharge, obtained a soluble sweet-tasting substance, and later, in 1784, he found that the same substance could be obtained from other oils, as well as from butter and lard. To this material he gave the name "the sweet principle of fats," and it afterward bore the name of Scheele's sweet principle or oil-sugar.

Lead plaster is said to have been discovered by the Roman physician Menecrates about the middle of the first century, and also to have been known to Pliny, who briefly described its uses, mode of preparation and application, but nothing was known of glycerin until Scheele's day.

Later the body was more carefully investigated by Chevreul, who determined its composition with tolerable exactitude and gave to it the name which it now bears. Pilouze in 1836 first established its formula. His experimental results corroborated Chevreul's views that the fats are ether-like compounds of the fatty acids. Henceforth glycerin became the subject of study by Berzelius, Liebig, Berthelot, and de Luca; but it remained for Wurtz to determine its exact chemical composition and relation to other bodies of the aliphatic series.

Scheele published the results of his investigations in a communication which appeared in the Transactions of the Royal Academy of Sweden in 1783.

He describes his method of preparation in the following terms: "It is not generally known that all solid oils obtained by pressure contain a natural sweet principle which differs in its special relations and properties from the other well-known saccahrine materials occurring in the vegetable kingdom. This sweet principle makes its appearance when oils of this kind are boiled with litharge and water until the whole of the litharge is dissolved by the oil. Water is then poured upon the 'emplastrum simplex' thus formed, the whole boiled for a few minutes, and on cooling the liquid is filtered off from the plaster and boiled until the residue becomes syrupy."

Glycerin was prepared by this process alone for many years, the lead introduced as an impurity from the litharge being removed before concentrating the filtrate by the use of sulphuretted hydrogen. This concentrated filtrate, after some primitive clarification, constituted the glycerin of commerce. Its rapidly increasing use soon demanded its production upon a larger scale. It was known in the process of soap-making that glycerin, liberated from the fat in the act of saponification, remained in the waste lye, but efforts to recover it from this medium with the extremely crude methods employed were unsuccessful. In the meantime the manufacture of stearin candles was undergoing important developments. Numerous patents were granted for processes for the decomposition of the fats and the separation of glycerin, as have been noted in the discussion of the manufacture of candle-stock.

In 1847 Sobrero in Paris discovered nitroglycerin. Alfred Nobel demonstrated its value as an explosive in 1863, and in 1866 invented dynamite. This was followed in 1875 by blasting-gelatin. The construction of vast engineering undertakings was greatly facilitated by the use of these substances, of which glycerin is the basis. The increased use reacted directly upon the source of the raw material and the soap trade soon came to a perplexing realization of the increasing value of the product being daily consigned to the sewer.

With the increasing demand for glycerin, the ingenuity of those most interested industrially was directed with renewed energy to its recovery from soap lyes.

During the past twenty years a great number of processes have been patented, all having a common object in the recovery of glycerin and salt. There can be traced a gradual evolution in efficiency of procedure and apparatus designed to remove the organic matter from the lye, to concentrate this clarified lye, thereby removing the greater part of the dissolved salts, and to continue the concentration until crude glycerin is obtained, which further remains to be purified by distillation.

A great variety of agents have been suggested for the removal of albuminous, resinous, and soapy matters; no less versatility has been displayed in the variety of methods for the separation of the dissolved salts; and in the concentration and further purification of the lye there has been apparently no limit to the ingenuity of engineers and chemists.

It is of interest to review briefly the history of the production and use of this commodity in the United States. It was first made in the United States in Philadelphia in 1847 by Robert Shoemaker. In 1848 the total amount of glycerin manufactured in this country was fifteen pounds. This was obtained by Scheele's original process from lead plaster. Domestic production in 1850 increased to an annual output of one hundred and fifty pounds. In 1856 glycerin was quoted at $1.75 per pound; to-day the prevailing market price is in the neighborhood of 15 cents per pound, and all soap makers' waste lye is treated for glycerin save where extremely limited production renders recovery uneconomical. The modern soap works has its glycerin plant, wherein are treated not only its own lyes but, where conditions make it possible, those purchased from competitors less favorably situated in this respect. The United States exports no glycerin, the home demand being sufficient to hold in domestic consumption practically the entire output, in addition to an annual European importation of upwards of fifteen million pounds chiefly of French origin. With the exception of the utilization of coal-tar, no industry presents such a complete triumph of applied science over empiricism as does the soap industry in the recovery of glycerin.

Glycerin Obtainable from Fats and Oils.—The more or less variable composition of fats and oils used as soap-stock, due to the varying proportions, according to their sources and methods of manufacture, of the different glycerides naturally occurring in them, forbid any exact statement as to the percentage of glycerin set free on saponification. The simplest index of the quality of any stock in this respect is the percentage of free fatty acids, it having been stated (page 124) that the presence of 10 per cent of free fatty acids is equivalent to a loss of about 10 per cent of the glycerin theoretically available. In the table on page 124 is indicated the percentage of glycerin liberated from the various glycerides occurring as constituents of commercial fats and oils. The percentage liberated in practice depends upon the quality of the glyceride body; and the percentage actually recovered, which is more variable, depends upon the manipulation during saponification and the efficiency of the subsequent treatment for its recovery from waste lye. It is commonly estimated in soap-manufacture that the following percentages of glycerin are liberated in the soap-kettle from the different stock mentioned:

Tallow....................	9.5 per cent.
Grease....................	9.0 " "
Cottonseed-oil.............	10.0 " "
Cocoanut-oil..............	12.0 " "

Palm-oil of good quality is comparable with grease; neutral olein stock, as olive-, corn-, and peanut-oils, is comparable with cottonseed-oil, and palm-kernel oil with cocoanut-oil. The percentage of finished glycerin obtained from waste lye is subject to great variation not only in different establishments according to the method of recovery used, but for different periods in the same establishment according to variation in the quality of stock used, its treatment in the kettle, and to the efficiency of methods employed for its recovery from waste lye.

Economic Position of Waste Lye.—Waste lye as a by-product in the manufacture of soap has become of increasing value with each succeeding year, not because of appreciation of the price of glycerin, for with increasing consumption of this commodity the price has declined without the depression of overproduction, but because of the imperative necessity on the part of the soap-manufacturer to recover in a once more than useless product an essential raw material of the industry and to produce as advantageously as possible a valuable article that may bear some of the burden arising from the shrinkage of profit on soap.

The commercial utilization of waste lye by individual soap-manufacturers depends primarily upon the quantity produced. With limited output and ready access to a refinery its sale to a competitor operating a plant for the recovery of glycerin is most economical. With larger output and under conditions peculiar to each works, the production of crude glycerin represents an advance in productive independence. The utilization of waste lye for the production of glycerin of dynamite refined quality is carried on most successfully by those manufacturers whose output or other sources of waste lye are sufficient to require not only the continual operation of the plant but its operation up to its fullest capacity. Fixed charges and depreciation of plant, the most important factors entering into the cost of glycerin, are thus reduced to a minimum.

Waste Lye.—In discussing the manufacture of settled soap we found the first product of the process was the stock lye and at a later stage the strengthening lye, which was worked over with fresh stock to remove the excess of caustic alkali. Where rosin is killed, as in the manufacture of rosined soap, an extra waste lye demanded consideration. The various methods of kettle practice, notably withdrawing the stock and rosin lyes with strength and then treating them with fresh stock for its removal; or withdrawing them neutral and discharging at once to storage-tanks; or withdrawing the stock lye neutral from stock killed with 15°–16° Bé.

caustic lye, the introduction of a pickle change to wash out the mechanically retained glycerin and the discharge to the sewer of a neutral rosin lye, were discussed. The relative advantages of the different methods of kettle practice become apparent on considering the by-products of soap-boiling from the standpoint of their utilization for glycerin. With the stock lye withdrawn with some strength which is not recovered, saponification may be complete, yet in the waste lye may remain a residual percentage of unused caustic which, although small, may amount in a year's operation to a very considerable quantity. Not only is there a direct loss of caustic, but the necessity of neutralizing the same with acid preparatory to the recovery of the glycerin present involves a double expense. In ordinary practice it is impossible to withdraw neutral lyes from settled soap made with the grade of caustic usually employed, viz., 74 per cent. The greater part of the alkalinity of the waste lye is due to the carbonate of soda which has been carried throughout the process as an inert and useless ingredient. It is true that carbonate of soda can combine with fatty acids present in all animal soap-stock, but that function is preëmpted by the more energetic hydrate.

With waste lye reduced to the lowest practicable degree of alkalinity, viz., 0.4 per cent (estimated as NaOH), it is transferred to storage-tanks to cool, whereby any soap dissolved may separate and heavy impurities subside, whence it may be drawn directly to the evaporator for concentration. Where waste lye is purified previous to evaporation, if free from soapy matter it may be treated at once and after filtration is ready for evaporation. The simple chemical and physical characteristics of a waste lye reveal as do those of no other product of the factory the quality of the raw materials used and the skill employed in their transformation.

Whether or not a waste soap lye is all that is to be desired depends entirely upon the point of view, whether we consider it as a waste product in the kettle-room or primarily as a raw material in the glycerin refinery. Moreover, the quality of the stock used in the kettle determines the quality of lye run off. From the soap-maker's point of view all lyes, except the strengthening lye, should be free from caustic alkali; should contain no soapy matter whatever;

should not contain an amount of salt in excess of that required to satisfactorily grain the soap, and should possess a color that indicates the thoroughness of the change from which the lye results as a purifying process.

The glycerin-refiner would be pleased to have a lye so free from organic impurities that glycerin foots would never disturb the even tenor of the refining process. But such conditions are impossible, the glycerin-refiner accepting such material as is delivered to him; while it is the constant endeavor of the soap-maker to eliminate from the soap throughout the process of boiling as much of the foreign organic impurities as is possible. In so far as the recovery of glycerin from such lye is concerned, it may be asserted as a general principle that the greater the inferiority of the stock used in the manufacture of soap, the greater will be the cost of the recovery of the glycerin present in the lye, not alone because of the presence of the high percentage of foreign organic matter necessary to be removed, but also because of the decreased yield of glycerin from such stock.

There are submitted in the following tabulation some characteristics of a number of waste soap lyes produced in as many soap works operating in the eastern United States. Their examination reveals diverse conditions of production, and especially discloses the fact that the smaller concerns manufacture in a manner contingent upon lack of skill and experience and an inferior quality of product.

These five lyes, whose characteristics are tabulated below, do not permit of themselves of deductions of general application, but they do in a measure serve to illustrate certain general ideas which familiarity with many other lyes proves in the majority of cases to be correct They have been arranged in the order of their color, the palest being first and each succeeding one possessing a deeper color than the preceding. Were the composition of the lyes uniform the color would be an index of the expense of the recovery of the glycerin. As the organic matter is removed from the lye with much more difficulty than the inorganic, a larger proportion in spite of the preliminary purification remains to constitute the "foots." It will be noticed that as the depth of color increases, the quality of the lye as a source of glycerin diminishes.

Naturally this observation does not apply correctly to a concentrated lye, but with unconcentrated lyes from settled rosin soap a statement of general application may be made to the effect that the color of the lye indicates the quality of the stock used, and as the color arises from the natural inferiority of the stock, it further, but less reliably, indicates that the darkest unconcentrated lyes possess the least value as sources of glycerin.

TABLE XXXIV.—COMPOSITION OF WASTE SOAP LYES.

No.	Per Cent NaOH.	Per Cent Na_2CO_3.	Per Cent NaCl.	Per Cent Glycerin.	Specific Gravity at 15° C.	
1	.15	.46	8.88	5.28	1.0878	Average 1.0956, Equivalent to 13° Bé.
2	.06	.73	12.04	5.05	1.0996	
3	.04	.09	10.46	4.70	1.0970	
4	.78	.79	8.76	1.28	1.0032	
5	.73	.85	7.12	4.04	1.1013	

The percentage of glycerin occurring in a waste soap lye is naturally affected by the dilution. Lye No. 1 of the above table is a high-grade soap lye, representing in its composition a high economy of operation with good native stock.

Lyes Nos. 4 and 5 contain an excessive quantity of free alkali; 0.73 per cent of NaOH represents a weight of 7641 pounds of 74 per cent caustic in 1,000,000 pounds of lye and is equivalent at the current price of caustic of this grade to a loss to the soapmaker of $308 per 1,000,000, pounds of stock. As conditions would hardly warrant the recovery of this percentage of alkali as soap, a direct outlay of not less than $113 for neutralization alone is made necessary in the initial step for the recovery of glycerin.

Yield.—In the manufacture of settled rosined soap the distribution of the total amount of lye produced during a boil depends upon factors so variable as to prohibit any accurate statement as to a definite percentage distribution. The volume may be estimated after each change by the depth of subsidence of the soap in the kettle. The percentage of the total lye withdrawn diminishes with each succeeding change.

With the highest economy of operation in the manufacture of settled rosined soap the minimum yield of waste lye per pound of glyceride stock is about 2.31 pounds, this yield being equivalent to 700 gallons per day from 1,000,000 pounds of stock per annum. This yield will be affected by the percentage of rosin used and the nature of the kettle practice.

The element of variability entering into calculations of this character is patent to every one possessing a knowledge of the industry, but with a product of uniform quality, conditions contingent upon production are sufficiently constant, especially when viewed through the perspective of a long period to make similar figures trustworthy for any particular establishment.

In the various transformations occurring in a boil of settled soap 1 pound of 74 per cent caustic will produce in waste lye an equivalent of 12 to 14 pounds. This is a proportion that likewise admits of extreme variability, not only with each works but with conditions prevailing therein during each boil.

Purification. Waste soap lye as run from the soap-kettle consists of water containing in solution glycerin derived from the fat from which the soap has been made, chloride of sodium which has been used for graining the soap, and a varying small proportion of free alkali, usually in the form of sodium hydrate or carbonate, and soapy, resinous, and albuminous matters, some of these latter being held in suspension as well as in solution. Various processes have been devised for removing the impurities and recovering the glycerin and salt as useful and valuable products from the soap lye. Such processes have consisted of steps relating on the one hand to the removal or neutralization of the free alkali present in the lye, and on the other hand to the removal of the organic impurities therefrom, leaving finally a purified solution consisting of water, glycerin, and salt, from which the water is eliminated by vaporization and the glycerin and salt thus recovered. The neutralization of the alkali has usually been effected by the addition to the lye of a suitable acid until the neutralization point is reached, and the product of this reaction remaining for the time being dissolved in the lye is afterward recovered therefrom in the form of a salt—for instance, chloride of sodium in the case where hydrochloric acid has been used for such neutralization, or as sulphate of sodium in the case of sulphuric acid having been employed.

To remove the organic impurities from the lye, recourse has been had to means for rendering such impurities insoluble, so that they could be removed by precipitation or filtration. The means employed to render these impurities insoluble have been, on the one hand, the addition of acids which threw them out of solution, and on the other hand the addition of certain metallic salts which were capable of forming insoluble metal-fat compounds, while in either case the albuminous impurities are coagulated and precipitated when the lye is brought to the neutral condition.

Procedure.—The impurities of waste lye comprise organic matter, chiefly albuminous matter from stock and highly colored resinous bodies from rosin, and inorganic matter as sodium hydrate and sodium carbonate, the former of which should have been completely removed in the soap-kettle and the latter representing impurity which is associated in varying degrees, according to its quality, with commercial caustic. If the lye has not been cooled and settled, more or less soapy matter will be in solution or suspension, or both.

Purification consists in neutralizing the alkalinity, decomposing the soapy matter, and coagulating the albuminous matter, for which purpose mineral acid, either hydrochloric or sulphuric, and crude basic persulphate of iron (chemical sand) are commonly used. The waste lye is tested for alkalinity, and if very slight, the iron salt alone may be used for neutralization; if considerable, the alkalinity may be reduced to a slight amount with acid and the remainder neutralized with the iron salt. The proportions of acid and iron salt used in neutralizing are three-fourths of the alkalinity to be neutralized by acid and the remainder by iron salt. The apparatus employed for purification is shown in outline in Fig. 204. It consists essentially of sheet-steel tanks of suitable capacity provided with open steam-pipes on the bottom for heating, a jet for the introduction of air for agitation, a tub for mixing the iron salt with water from which it is discharged by gravity into the mixing-tank, a pump for forcing the heated lye treated with iron salt and acid through the filter-press, from which the filtrate flows into the adjacent tank. The process of purification is completed by the use of another tank of corresponding capacity surmounted by a second but smaller filter-press. As a result of the first treatment the alkali is neutralized, and converted into chloride and sulphate according as the acid used is hydrochloric or sulphuric; soapy matter has been decomposed and albuminous matter coagulated. During mixing and filtration the lye is heated and agitated, whereupon filtration being complete, the impurities remain as solid cakes in the filter-press, and the filtrate clear, and if possible colorless, in the adjacent filtrate-tank. The filtrate should be very slightly acid, which is neutralized by the addition of dry soda-ash in calculated amount and thoroughly mixed, whereupon the lye is again filtered and should flow from the press, clear and colorless, free from organic matter removed by the first treatment, and free from soluble iron salts and any soluble coloring matter that may have escaped the first treatment. The purified waste lye is now ready for evaporation. An arrangement of waste-lye treatment-tanks with filter-press is shown also in Figs. 207 and 216. In Fig. 35 is shown a similar equipment designed for the filtration of cottonseed-oil.

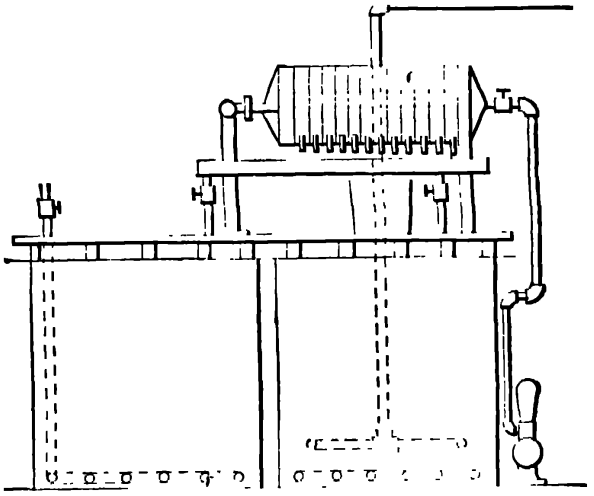


FIG. 204.—Arrangement of Waste-lye Treatment-tanks.

Instead of the basic ferric sulphate (iron salt) any of the alums may be used as the coagulant. The iron salt commonly called "chemical sand" is made essentially as follows: An iron ore analyzing 50 per cent of iron oxide, or its equivalent of alumina, is dried, reduced to powder and mixed with proper proportions of 60° Bé. sulphuric acid previously heated to about 300° Fahr. After thorough and rapid mixing the mass is dumped from the mixing vessel upon an iron floor where the reaction continues and the mass solidifies;

whereupon it is broken up and heated in a revolving drum at a temperature of 380° to 500° Fahr. During the first treatment the

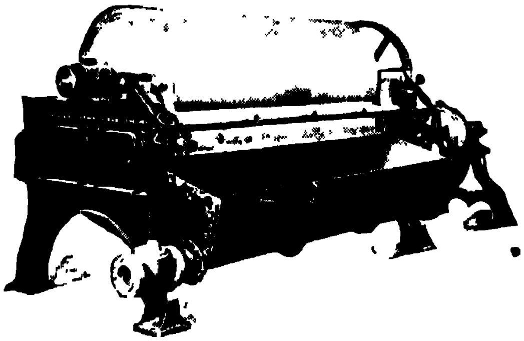
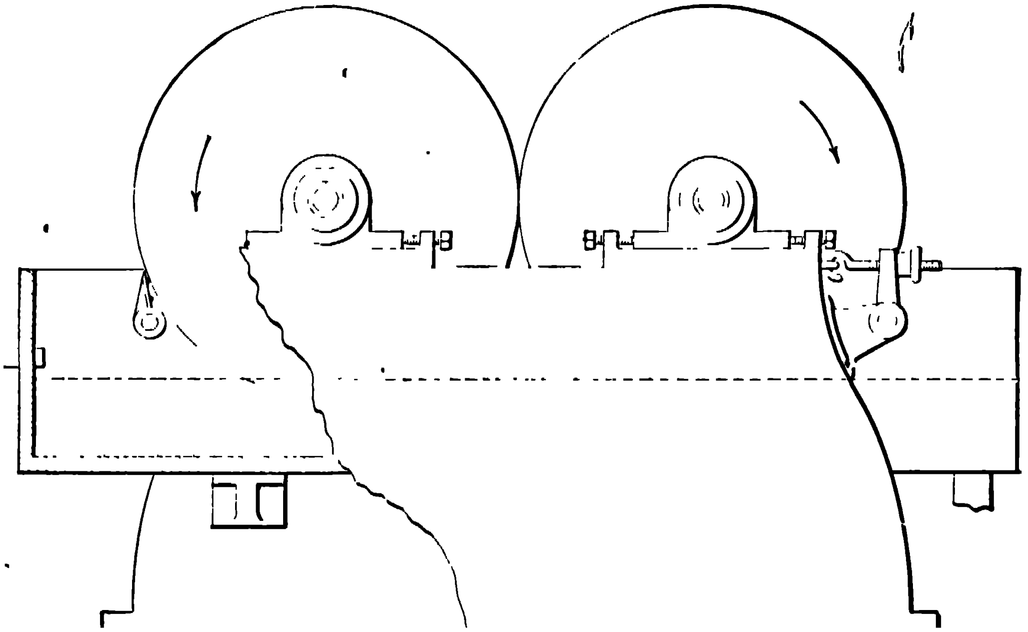


FIG. 205.—Single-roll Film Evaporator.

iron ore and acid enter into partial combination; during the second treatment a persulphate is formed by combination of the free ore partly with free acid and partly with the acid sulphate formed during the first treatment. During the second treatment heating is continued for several hours, according to the temperature and quantity of the mass treated. The product is a somewhat basic persulphate of iron, with more or less free acid, and is readily pulverized by reason of its softness.

Concentration under Atmospheric Pressure.—Concentration of waste lye may be effected in open tanks provided with a closed coil wherein either live- or exhaust-steam may be used. This procedure is the least efficient of all methods of evaporating the surplus water from waste lye. The degree of concentration is determined primarily by the steam pressure available and can never economically be carried very far. Where the volume and quality of waste lye and cost of fuel warrant, an advance in efficiency would be represented by the use of a film evaporator, single- and double-roll types of which are shown in Figs. 205 and 206, and which consists essentially of a shallow iron vat on which are supported respectively one and two hollow cylinders, geared in the latter case to revolve in opposite directions, as indicated by the arrows. Steam is suitably admitted into both cylinders and the level of waste lye is constantly maintained, as indicated by dotted line. The liquor is drawn up and over the cylinders in a thin film, and when saturation ensues solid matter is removed from them by means of scrapers. The concentrated product is drawn off by gravity from the bottom of the vat. As evident from the construction of this type of evaporator, separation of salt and further concentration of the product cannot proceed satisfactorily.



56. Doub [illegible] Evaporato

Principles of Evaporation.—Pure water at 100° C. (212° Fahr.) boils, or gives off vapor of the same pressure as that of the surrounding atmosphere. At atmospheric pressure the boiling point will rise according to the amount of matter in solution and will correspondingly increase as the density of the liquor increases from loss of water by evaporation, requiring therefore not only a greater amount of heat to raise the temperature of the solution to the boiling point than when pure, but increasing application of heat to maintain the solution at a boiling temperature as its density increases from evaporation. Regnault estimated that the quantities of heat contained in equal weights of water and air at the same temperature are in the ratio of 4.21 : 1.00; or that the heat which is liberated when water cools down to 100° C. (212° Fahr.) is sufficient to raise the temperature of 4.21 times as much air to the same extent. In passing into the state of vapor water absorbs 5.36 times as much heat as is required to raise its temperature from 0° C. to 100° C. (32° to 212° Fahr.), and this quantity of heat becomes latent, i.e., it produces no increase of temperature in the steam, and therefore is not indicated by the thermometer and becomes sensible only when the vapor is condensed. Thus one pound of steam at 100° C. (212° Fahr.) in condensing to form boiling water will yield sufficient heat to raise the temperature of 5.36 pounds of water (or 4.21 × 5.36 = 22.6 pounds of air) to 212° Fahr.

Various devices have been introduced to utilize most efficiently this convenient store of heat. The influence of reduced pressure upon the boiling-point of water is shown in the following table:

The temperature of water boiling at atmospheric pressure is 212°
“ “ “ “ “ under 5″ vacuum is 195°
“ “ “ “ “ “ 10″ “ 185°
“ “ “ “ “ “ 15″ “ 160°
“ “ “ “ “ “ 20″ “ 150°
“ “ “ “ “ 25″ “ 130°
“ “ “ “ “ “ 26″ “ 120°
“ “ “ “ “ “ 27″ “ 112°
“ “ “ “ “ “ 28″ “ 100°
“ “ “ “ “ “ 29″ “ 72°
“ “ “ “ “ “ 29½″ “ 52°

Other liquids follow a similar rule but have different normal boiling-points. Thus it is clear that in a single vessel in which varying degrees of vacuum are maintained aqueous solutions may be boiled and thereby concentrated with increasing efficiency as the vacuum is increased. It is likewise apparent from these facts that if in several vessels there are different degrees of vacuum produced, we can have a descending scale of boiling temperatures so that vapor of comparatively low temperature can be utilized as it is produced. The different boiling points of a liquid under different pressures can be directed toward greater efficiency than is possible in a single vessel by making the vapor given off in boiling the contents of the first vessel at a certain pressure form the heating agent of the liquid boiling in the second vessel at a lower pressure, the vapor from this second vessel forming the heating agent of the third, and so on.

This principle governs all forms of what are known as multiple effect evaporators. In such an apparatus the latent heat of vapor of liquids boiling under a low vacuum is used to boil a second vessel working under a higher vacuum. The usual limit to this system is four pans, thus utilizing latent heat four times and reducing the fuel to nearly one-fourth of that required for open evaporation, or single effects.

With liquids liable to injury by heat the total variation in temperature available under ordinary conditions is that between the temperature of steam at 5 pounds pressure (227° Fahr.) and the temperature of a solution at 30° Bé. boiling under a vacuum of 26″ (131° Fahr.)—a total of 96° Fahr. With a triple effect, or three vessels, there is steam at 5 pounds pressure in the drum or shell of the first effect and the liquid in the tubes (at atmospheric pressure) boils at 227° Fahr., giving off vapor at 212° Fahr. This vapor at 212° Fahr. passes into the shell of the second effect and boils the liquid in the tubes of this effect under 14″ vacuum, giving off vapor at 161° Fahr. This again passes to the shell of the third effect and boils the liquid in the tubes under a vacuum of 26″, the boiling-point of this liquid (supposing it to be a concentrated solution of 30° Bé.) being 131° Fahr. It is a point worthy of note that when the liquid is a solution of solids in water the vapor will always be at the temperature of boiling water at the pressure to which the liquid is subjected at the time, while the liquid itself will be slightly warmer; the total difference of temperature in concentrating liquids liable to injury by heat being, as already mentioned, 96° Fahr. While the amount of heat transmitted through the tubes (and therefore the work done) is practically proportional to the difference in temperature, it is evident that the same work is done whether the whole of this difference is in several vessels or in one vessel. In other words a double, triple or quadruple effect can only do the work of a single effect the size of the first vessel of the multiple effect, but it does it with one-half, one-third or one-quarter of the quantity of steam or fuel respectively.

Vacuum Evaporators for Waste Lye.—Evaporators designed for the concentration of waste lye *in vacuo* by means of steam as a heating agent may be divided according to the number of vessels in which vacuum is maintained and evaporation takes place into single effects and multiple effects. Multiple effects of more than two vessels for the concentration of waste soap lye have not been demonstrated to have any materially greater efficiency. According as the vessel containing steam surrounds the vessel in which vacuum is maintained and evaporation takes place, or is in the vessel in which vacuum is maintained and is surrounded by the liquid to be concentrated, single-effect evaporators may be divided into two general classes. The first class is represented by a steam-jacketed and insulated evaporator shown in Fig. 207. The second class of evaporators, the distinctive feature of which is the introduction of the steam-chamber into the vessel in which vacuum is maintained and which is surrounded upon all sides by the liquid to be concentrated, may be subdivided into two classes, according as heat is applied, in the form of steam from the outside or inside surface of the tubes of the steam-chamber. Evaporation from the outside surface of the tubes of the steam chamber is represented in the type shown in Figs. 208 and 209 which is well adapted for arrangement in multiple effect for the concentration of purified glycerin and caustic-soda solution; in the latter case when the soap-manufacturer prepares his own caustic soda by the causticization of soda-ash. For this purpose all the forms of evaporators described are equally well adapted. Evaporation from the inside surface of the tubes of the steam-chambers is represented by the Foster (Fig. 210) and Jobbins (Fig. 211) evaporators.

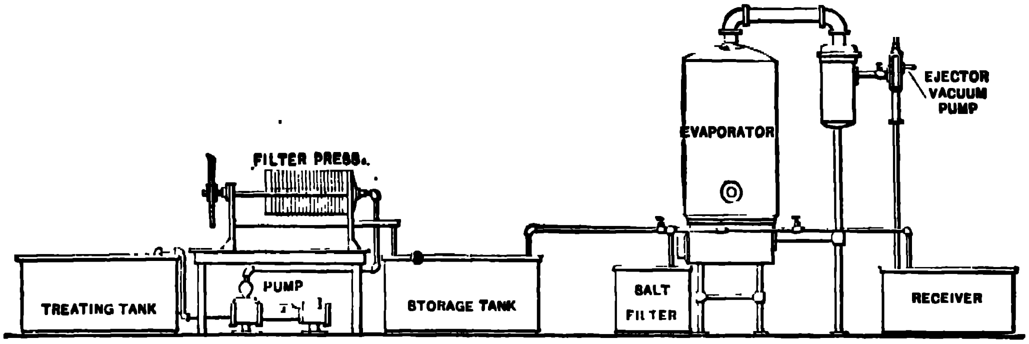


FIG. 207.—Crude Glycerin Plant with Steam-jacketed Evaporator.

Evaporating-plant.—The mechanical equipment of a plant for the concentration of waste soap lye comprises essentially an evaporator, which may be of any of the types mentioned; a supply of water for the condensation of vapor and its removal either by a simple ejector (Fig. 207) or by a wet-air vacuum-pump; tanks for the treatment of waste lye; a filter-press and a storage-tank for the clarified filtrate; tanks for the storage of the concentrated product, called in its final concentrated form crude glycerin; a suitable filter for the separation of the solid and liquid products discharged from the evaporator; suitable pumps for the agitation of waste lye, for forcing it through the filter-press and for operating the salt-filter located to one side of the evaporator and beneath its

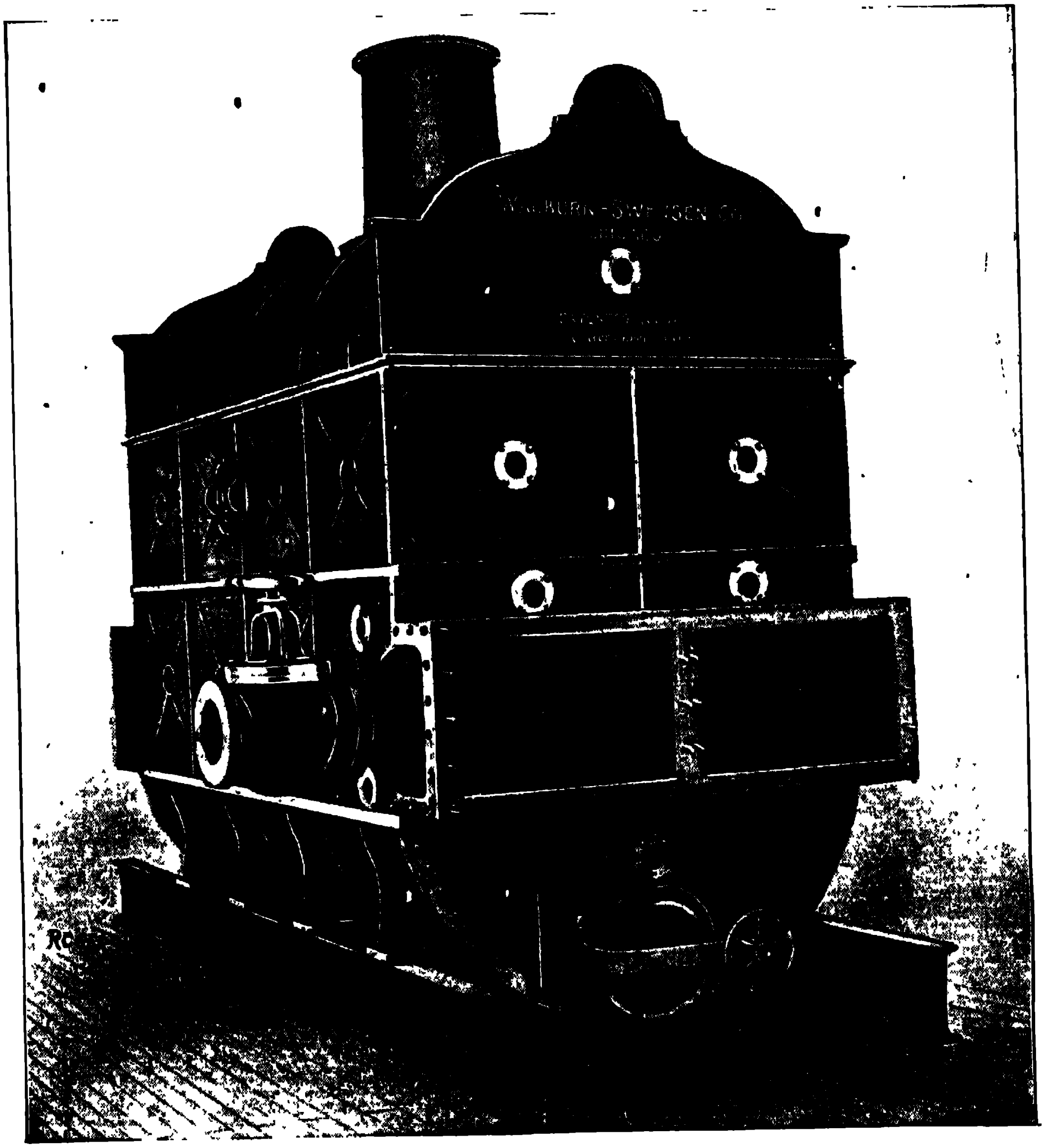


FIG. 208.—Evaporator for Waste Soap Lye—Walburn-Swenson.

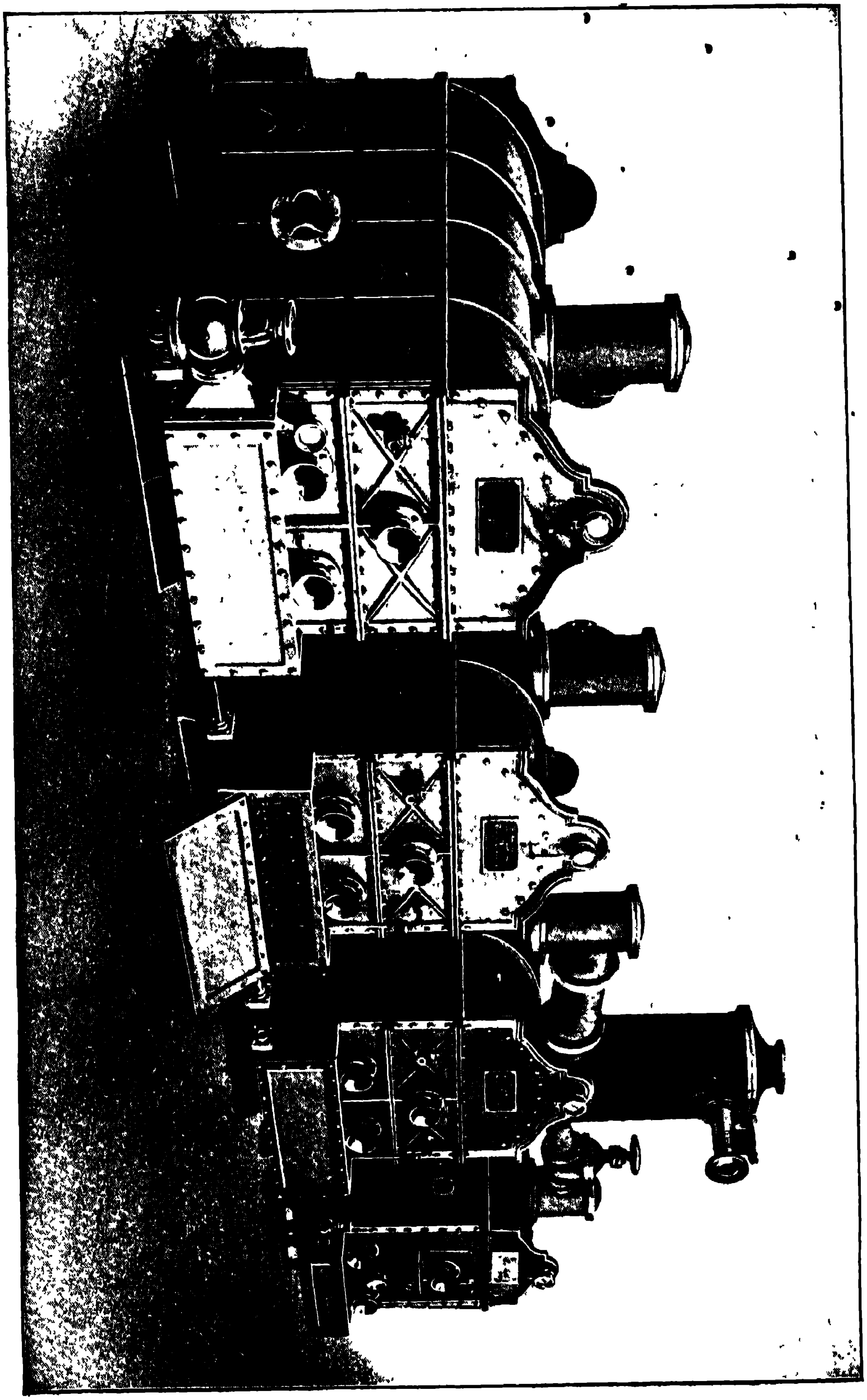


FIG. 209.—Triple- and Single-effect Evaporators for Glycerin and Caustic Soda.

discharge door; and finally steam at 80 pounds pressure for heating the waste lye preparatory to filtering, for operating the air-pump for the agitation of the waste lye, for operating the filter-press pump, for supplying heat to the evaporator, and for operating the vacuum-pump and the suction-pump of the salt-filter. The procedure of operation varies not only with each type of evaporator, but to a greater or less degree in each plant wherein an evaporator of the same type is installed, according to the steam pressure available, the quantity and purity of the waste lye and the judgment and experience of the attendant.

Foster Waste-lye Evaporator.—This type of evaporator for concentrating and recovering crude glycerin from waste lye and recovering the salt from the same during concentration by single or multiple-effect apparatus working under vacuum is shown in detail in Fig. 210.

The improvements comprise a combination of evaporating-plant and drawing-off valves and filtering and drying appliances.

Fig. I of Fig. 210 represents a sectional side elevation, and Fig. II an end view of the concentrating apparatus, while Figs. III and IV are horizontal sections as taken on the lines 3–3 and 4–4, respectively, of Figs. I and II.

Referring to the various figure numbers of Fig. 210, the evaporating-vessel *F*, into which the lyes to be treated are supplied, is fitted with a steam-drum *F′*, in which are tubes for the liquor to circulate through; the bottom cover F^2 of the evaporator is formed of a downwardly-sloping angular shape toward one side, so that the salts which are precipitated are swept down into the filter *A* by the rapid circulation of the liquor under treatment. Ports F^3 are formed on the heating-chamber of the evaporator, and the vapor-supply pipe *J* is carried down to about the centre level of the heating-chamber, by which a most perfect distribution of the vapor and circulation of the liquor under treatment are obtained.

A valve-chest *B′* is fitted with a valve *B* and is located between the bottom cover F^2 of the evaporator and the filtering-vessel *A*, and two of these are preferably used in connection with each evaporating vessel, so that the one is alternately shut off while the other is working. This filtering-vessel *A* is fitted with a sight-glass *E* at its one end to see when the filter is full of salt and with a door *I* at its other end for removing the dry filtered salt. A steam-valve *H* is fitted for admitting steam to the perforated tube *G*, and an air-valve *D* and safety-valve *D′* are also fitted on top, while a cock *C* is fitted from whence a pipe connection *C′* would be taken to the upper vacuum space of evaporator *F*. The filter is fitted with a filter-bed *A′* of iron netting and copper gauze and an iron grid-plate.

Operation.—In working the salt-filters when the apparatus is started to work, the valve *B* at the bottom of the vessel connected to the salt-filter is opened (and that in the other filter shut) and during the concentration of the liquor the salt that is formed is precipitated into the salt-filter *A* and when filled with salt, which can be seen in the sight-glasses *E*, the valve *B* is shut (and the valve *B* on the other salt-filter is opened), and cock *C* on the pipe *C′* is then opened to the vacuum space in the top chamber *F*. The steam-cock *H* is opened at the same time as cock *C*. The steam from cock *H* passes through the perforated pipes *G* and drives the liquor through the salt, the liquor and steam being sucked through cock *C* up into the top chamber *F* by the vacuum. When the salt is thoroughly dried, which can be seen in the sight-glasses *E*, cocks *H* and *C* are shut and the air valve *D* opened, and door *I* is opened for taking out the dry salt. After the salt is taken out the door *I* is closed and cock *H* opened, and when the steam has blown all the air out of the salt-filter through cock *D* both cocks *H* and *D* are shut and valve *B* is opened, gently at first, to allow the steam in the salt-filter to pass quietly into the apparatus. After the steam has all passed and the liquor is seen to rise in the sight-glasses *E* valve *B* is opened full. These salt-filters are worked alternately. In small apparatus only one salt filter is used, there being placed between the salt-filter *A* and the bottom cover F^2 of the vessel *F* a salt-hopper to receive the salt precipitated during the operation of drying in the salt-filter.

Jobbins Waste-lye Evaporator.—The dominant system of glycerin recovery from waste soap lye throughout the world is that covered by the patents of Jobbins and Van Ruymbeke, and represents in ease and simplicity of operation and in quality of product the most successful and practicable solution of this long-baffling

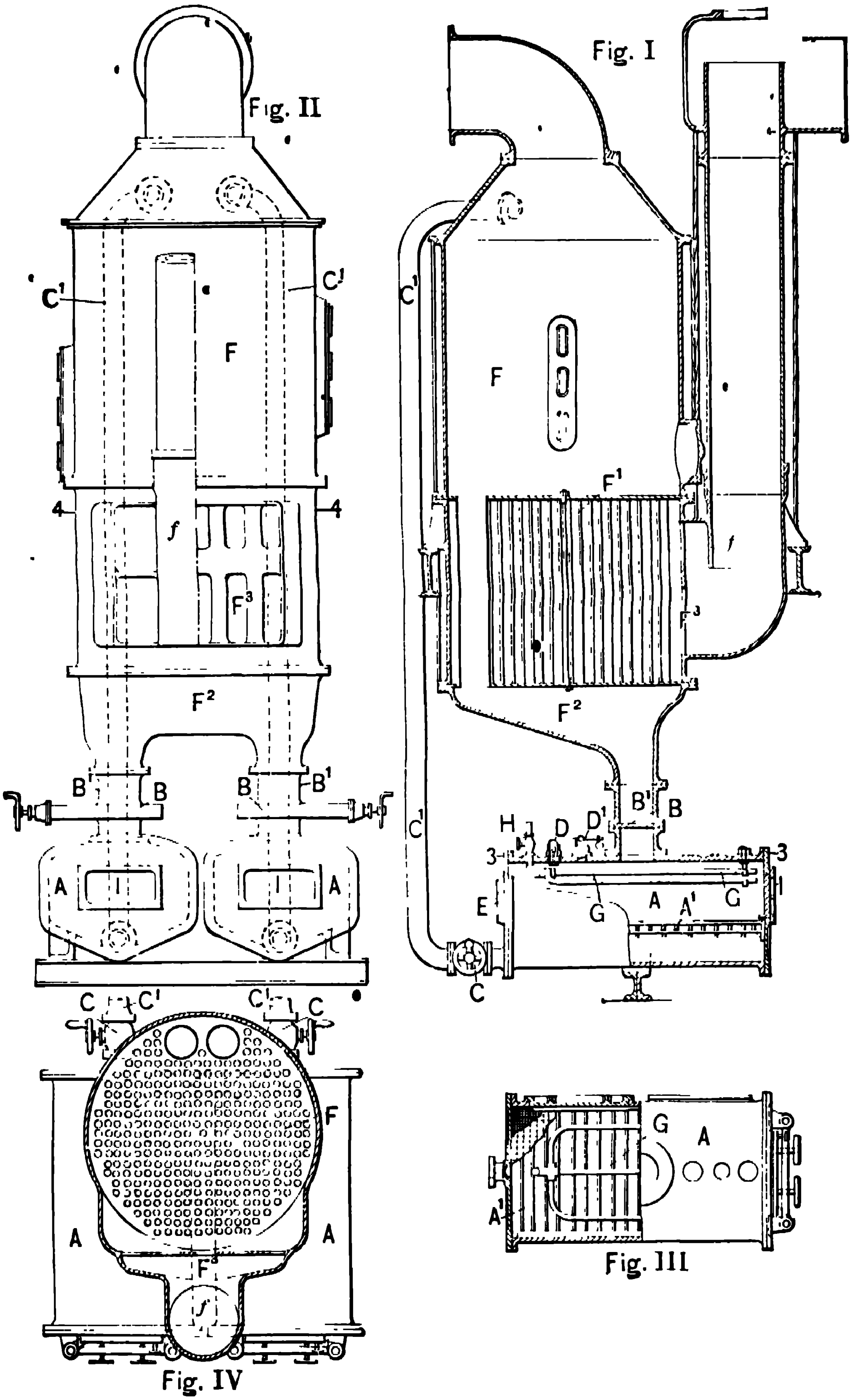


FIG. 210.—Foster Waste Soap Lye Evaporator.

problem of chemical engineering. As a result of the increasing demand for glycerin and for products made from it during the past half century great ingenuity has been directed to its recovery from waste lye where since Chevreul's researches it had been known to exist.

During the past twenty-five years a great number of processes have been patented, all having a common object in the recovery of glycerin and salt. There can be traced a gradual evolution in the efficiency of procedure and apparatus designed to remove the organic matter from the lye, to concentrate this clarified lye, thereby removing the greater part of the dissolved salts, and to continue the concentration until crude glycerin is obtained which further remains to be purified by distillation.

A great variety of agents have been suggested for the removal of albuminous, resinous, and soapy matters; no less versatility has been displayed in the variety of methods for the separation of the dissolved salts; and in the concentration and further purification of the lye, there has been apparently no limit to the ingenuity of engineers and chemists.

The Jobbins system of glycerin recovery comprises the purification of the waste lye, the use of an improved evaporator for its concentration and the separation of salt, and the use of improved apparatus and the application of agents in a new way to the distillation of the concentrated product.

Construction.—Referring to Fig. 211, 1 and 2 represent cylindrical evaporators made of sheet-iron; 3, 3, 3, are vertical posts or pillars arranged in pairs and of the proper height and 4, 4, are supporting braces at the top of the pillars for supporting the evaporators 1 and 2. 5 and 6 are circular heads or flue-sheets within the evaporator 1, and 7, 7, are flues connecting the heads. 8 is a steam-inlet pipe for admitting steam into the space between the heads 5 and 6 which constitutes the steam-drum, this pipe having a valve 9; 10 is an outlet pipe for condensed steam from this space, having a valve 11. 12 is a water-gage or column on the outside of the evaporator, connected with the steam-drum, for measuring the amount of condensation therein. 13 is a manhole or door in the lower part of the evaporator for removing the salt or other deposits therefrom, and 14 is a similar door in the evaporator about two-thirds of the way from the bottom thereof, to allow the interior of the evaporator to be reached for the purpose of cleaning. 15, 15 are draw-off pipes provided with valves 16, 16, and connected with the pipe 17. Use is made of two or more of these pipes arranged one above the other, in order that the liquid may be drawn off close to the upper layer of the salt deposited therein. 18 is a gate valve at the bottom of the evaporator for drawing off the contents thereof for any desired purpose. 19 is a vacuum draw-off pipe connected with the interior of the evaporator above the maximum limit of the deposited salt. 21 is a valve in the pipe from the evaporator which connects with pipe 19, and 22 is a valve at the bottom of this pipe. By opening the valve 21 a portion of the liquid in the evaporator may be run out into the pipe 19, after which the valve 21 is closed and the valve 22 is opened and the small quantity of liquid in the pipe 19 is removed and may be tested. In this way a sufficient quantity of liquid in the evaporator may be drawn out for the purpose of testing, against the vacuum therein, and without affecting the vacuum. 23 is a water-gage on the side of the evaporator and connected therewith for determining the height of the liquid therein. 24 is a steam-gage on the front or side of the evaporator, connected with the steam-space between heads 5 and 6, for determining the pressure therein, and 25 is a vacuum-gage for indicating the degree of vacuum in the evaporator. 26 is a pipe leading into the evaporator and provided with a valve, by means of which lye not saturated with salt may be introduced into the evaporator for the purpose of dissolving out any salt which may accumulate in the flues 7 and around the same, and thereby prevent the incrustation thereof. Waste lye should be allowed to remain in contact with the pipes to be cleaned for a sufficient length of time to dissolve the incrusting matter. 27 is a pipe with valves, opening into the evaporator above the steam-drum therein, for introducing the material to be evaporated. 28 is an annular plate within the evaporator near the top thereof, but of less diameter than the same, strengthened by braces 29 and having the hole or opening 32 at its central part. 30 is a circular dash-plate of larger diameter than the opening 32 and supported directly beneath the same by rods 31. This latter dash-plate 30 serves to prevent in a great measure any of the material from being projected from the evaporator in case of a sudden application of vacuum or steam heat. 33 is a heavy vapor-pipe leading from the evaporator and provided with a gate-valve 34 therein. 35 is a pipe from the evaporator 2, provided with a gate-valve 36 therein the latter being arranged

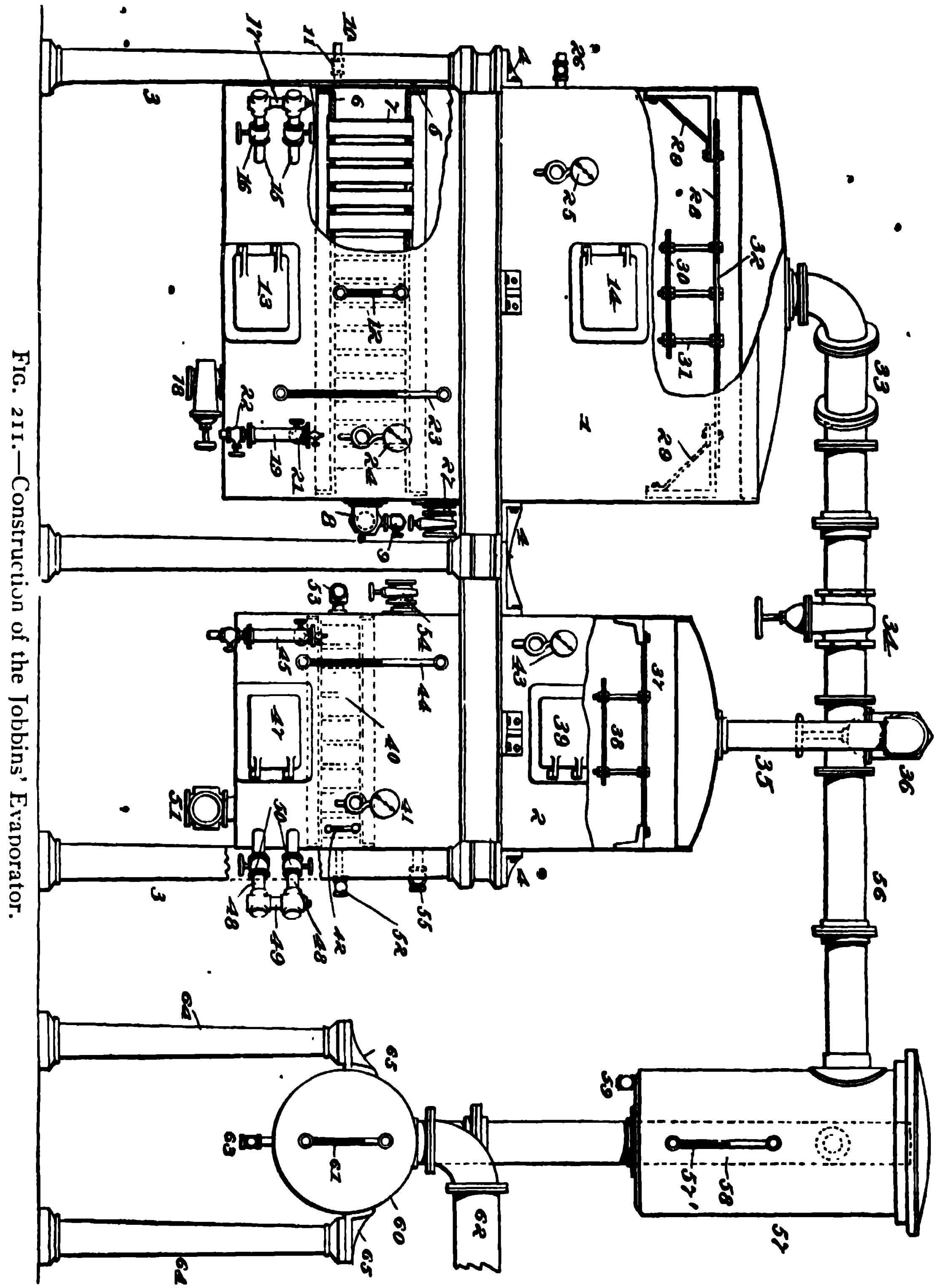


FIG. 211.—Construction of the Jobbins' Evaporator.

above the pipe 32, so that any material from the evaporator 1 will be prevented from entering the evaporator 2. 37 is an annular dash-plate in the evaporator 2, and 38 is a circular dash-plate secured beneath the same. These elements correspond to those in the evaporator 1. 39 is a manhole for entering the evaporator 2. 40 is a steam-drum, 41 a steam-gage connected therewith, 42 a water-gage or column for determining the amount of condensation in the steam-drum 40. 43 is a vacuum-gage and 44 a water-gage for determining the height of the liquid in the evaporator. 45 is a vacuum draw-off pipe on the evaporator, corresponding to the pipe 19 of the evaporator 1. 47 is a door at the lower part of the evaporator for removing the salt and other sediment that may be deposited therein. 48, 48, are draw-off pipes for removing the contents of the evaporator close to the salt therein, connected with the pipe 49 and provided with valves 50. 51 is a draw-off pipe corresponding to the draw-off pipe 18 of the evaporator 1. 53 is a steam-pipe for admitting steam into the steam-drum 40, and 52 is a pipe for removing the water of

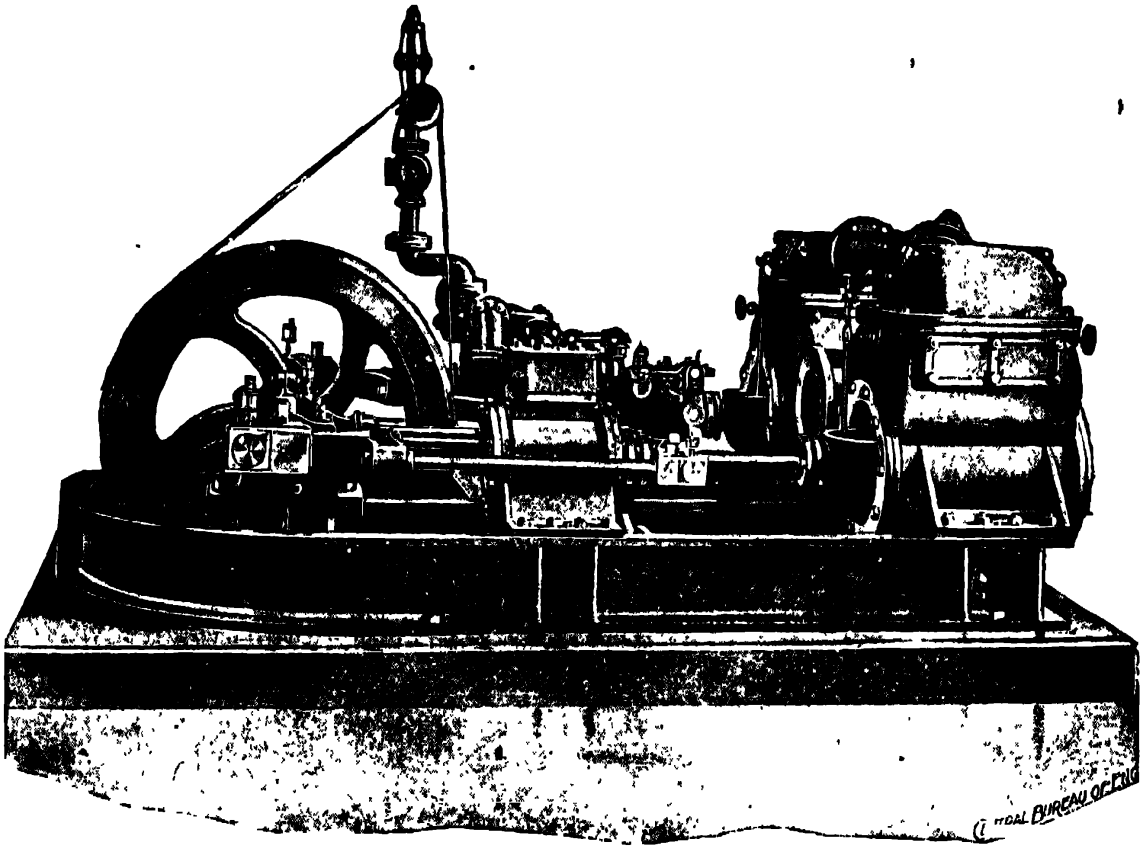


FIG. 212.—Duplex Fly-wheel Vacuum-pump. Size, 10 × 16 × 10.

condensation therefrom. 54 and 55 are pipes on the side of the evaporator for introducing the material therein. 56 represents the continuation of the pipe 33 leading into the catch-all 57. Within this catch-all 57 is a pipe 58, its upper end being open and arranged with its opening above the upper level of the pipe 56 passing down through the bottom of the catch-all 57 and connected with the drum 60. 59 is a draw-off pipe provided with a valve therein for removing any condensed liquid or material entrained or mechanically carried off with the distillate that may be deposited in catch-all 57. The catch-all 57 is provided with a removable head and with a water-gage 57′, for determining the amount of material that may be caught by and deposited within the same. 60 is a horizontal cylindrical drum beneath the catch-all 57 and connected with the pipe 58, as before stated. This drum 60 is provided with a water-gage 61 for indicating the amount of condensed water therein. 62 is a pipe extending up from the top of the drum 60 and connected with any desired form of vacuum-pump. 63 is a draw-off pipe for removing the contents of the drum 60, either into a tank placed beneath said drum or to any desired receptacle. The drum 60, on which rests the catch-all 57, is supported on the standard 64 by means of lugs or brackets 65 bolted to the tops of said standards and to the drum.

Vacuum is maintained in the evaporating system usually by means of a wet-air pump of the ordinary type permitting a working vacuum of from 25 to 27 inches. In the distilling system vacuum is maintained by a pump of the duplex fly wheel type, one form of which is shown in Fig. 212.

Concentrated Waste Lye.—Concentration of waste lye may be either partial or complete, according to the steam pressure available and the character of the apparatus employed, and in general is determined by the extent of manufacturing operations and volume of waste lye produced. The product of partial evaporation is simply concentrated lye; of complete evaporation, crude glycerin, the two products differing in the proportions of water, salt and glycerin present. With inadequate steam pressure the production of concentrated lye of a quality approaching crude glycerin may be effected in the following general manner: During the first day the evaporator is fed with waste lye; the concentrated product is discharged at night and the salt separated. During the forenoon of the second day the evaporator is fed with waste lye, and in the afternoon the concentrated product of the first day's work is drawn in as required. At night the whole concentrated product is discharged and the salt separated as before. During the forenoon of the third day the evaporator is fed with waste lye, and in the afternoon the concentrated product of the first and second day's work is drawn in as required. The whole concentrated product of three day's work is discharged at night, and, according to the steam pressure available, a fair quality of crude glycerin may be obtained. Wherever practicable each period of evaporation should be started with waste lye, thereby clearing the tubes of the steam-chamber from adhering salt and permitting more efficient utilization of the steam available.

Operation.—The operation of the complete evaporating system designed for the production of crude glycerin suitable for immediate distillation comprises the use of two evaporators shown in relative size, position and with connections in Fig. 211. Suitable connections at pipe 62 for water for the condensation of vapor and for the pump for the removal of vapor from the evaporating system, as well as discharge-tanks of suitable size located beneath their respective evaporators and also salt-filters located below and in front of the discharge doors of their respective evaporators, are not shown in the figure. In operating evaporator 1 condensing water is admitted to the vacuum-pump in proper amount, whereupon the pump is started. Waste lye purified, as already described, is introduced into evaporator 1 through the pipe 27 which connects with the storage-tank for waste lye prepared for evaporation. Valve 36 is then closed, in event of evaporator 1 being operated alone, so as to maintain a fairly high vacuum, preferably say about 26 inches, within the drum 60, catch-all 57 and evaporator 1. Steam is now introduced through the pipe 8 into the steam-drum of the evaporator which heats the flues 7 and the heads therein tothe proper temperature. This heat should be sufficient to concentrate the lye until it has been concentrated to the proper degree, preferably to about 28° Bé. when drawn from the evaporator, or 30° Bé. at 15° C. This heating may be conveniently accomplished by means of exhaust-steam. This evaporation of the lye causes the salt therein to separate after the salting-point has been reached, which salt accumulates at the bottom of the evaporator and may be removed therefrom through the door 13 and dried upon the salt-filter described later.

By means of the pipe 19 the specific gravity of the lye may be tested, and when it has reached the desired strength, preferably that indicated, the lye is drawn off as closely to the salt as possible through one of the draw-off pipes 15. By making use of the annular and circular dash-plates 28 and 30, the liquid is prevented from being projected into the pipe 33 by any sudden increase of the vacuum or steam-heat. At the above described density (30° Bé. at 15° C.) the lye contains about 50 per cent of glycerin and most of the salt contained therein has been deposited.

By carrying on the evaporation in a vacuum of about 26 inches and with steam at a low pressure, the loss of glycerin by evaporation is reduced to a minimum, and in fact there is practically no loss of glycerin from this source when the process is properly carried out.

The product of evaporator 1 may be called half-crude glycerin and is a concentrated lye of 30° Bé. at 15° C. This having accumulated in sufficient quantity is subjected to further concentration in evaporator 2, removal thereinto being generally accomplished by the suction of the high vacuum therein, by means of steam introduced into the steam-drum 40 until the lye has attained a specific gravity of 34° Bé. at 15° C. An additional quantity of salt separates during this operation, which salt may be removed and dried in the salt pan attached to evaporator 2, called from its product the crude evaporator. The reason for carrying on the evaporation process in two steps as above described, is that when the salt is impregnated with concentrated crude glycerin it is difficult to dry and to free from its adherent crude glycerin, but by evaporating it in the first evaporator until it reaches a specific gravity of not more than 30° Bé. at 15° C., and discharging the contents and separating and washing the salt, and further concentrating the crude liquor and again removing salt, this disadvantage is reduced to a minimum. In shutting down the evaporators, whether the half-crude or crude, the heating steam is first turned off; next the condensing water to the vacuum-pump and then the pump. The vacuum is now broken and the contents discharged.

The product of the last evaporation is crude glycerin or a saturated solution of common salt, in glycerin, together with a small portion of impurities. The product is now ready for distillation.

The variation in the composition of purified waste lye as it undergoes progressive concentration to the state of crude glycerin is shown in the following analytical determinations made by Gill and Miller on products obtained from waste lye purified by treatment with sulphuric acid and persulphate of iron:

TABLE XXXV.—OBSERVATIONS OF WASTE LYE SAMPLES AT SUCCESSIVE STAGES OF CONCENTRATION.

Sample.	Specific Gravity, Westphal Balance.	Per Cent of Salt.	Per Cent of Sulphate, Na_2SO_4.	Per Cent of Glycerin.	Specific Heat.
A	1.120	11.1	2 3	6 2	0.8233
B	1.163	12 5	not determined	8.0	0.7841
C	1 206	16 4	4 2	8.1	0.7488
D	1.243	18 0	2 0	16.4	0 7313
E	1.204	0 4	0 4	55.5	0 6030
F	1.330	5 4	very small	87.0	0 4960

Determinations of boiling points, under atmospheric pressure, of concentrated lyes of different proportions of glycerin and salt were made by the same investigators as follows:

TABLE XXXVI —BOILING-POINTS OF WASTE LYE WITH CORRESPONDING PERCENTAGES OF GLYCERIN AND SALT.

—	Boiling-point.	Glycerin.	Salt.
	°C.	Per Cent.	Per Cent.
1	100	16 10	20.21
2	not taken	17.30	20 02
3	112	32 86	17.05
4	120	63.28	10 74
5	240*	87.84	6.34

* Taken at a point where ebullition had just ceased. The lye was boiled down in the open air, and samples taken for analysis as the boiling-point was noted.

Products of Concentration.—The products of concentration with evaporator 1 are half crude glycerin and salt; with evaporator 2, crude glycerin and salt. Where sulphuric acid is used to neutralize the alkalinity of waste lye, it has been found that in evaporating these lyes, the first salt which separates after the salting-point has been reached consists essentially of sodium sulphate, often to the extent of 80 per cent, the remaining 20 per cent consisting of sodium chloride or common salt. Therefore, when it is desired to manufacture Glauber's salt or to obtain the salt as free as possible from the sulphate, the evaporation is stopped when most of the sulphate has separated; and the crude sulphate thus obtained is purified by solution in water and crystallization such operation being repeated if necessary until sufficiently pure Glauber's salt is obtained. After the separation of the impure sodium sulphate as above described, the evaporation of the liquid is continued until it reaches a density of 28° Bé. as drawn from the evaporator or 30° Bé. at 15° C. Where the separation of the sodium sulphate is not desired, the evaporation may be carried on to this point continuously from the beginning. At the above described density (30° Bé. at 15° C.) the lye contains about 50 per cent of glycerin, and most of the salt originally contained therein has been separated.

In discharging the contents of either evaporator the hot liquor is run by means of suitable outlets into storage-tanks beneath the respective evaporators. Discharge door 13 or 47, whether the half-crude or crude evaporator is being operated, is opened and the remaining liquor and salt are scraped into the salt-filter. This consists essentially of a shallow tank of suitable capacity provided with a false bottom or screen a couple of inches above the bottom and which is covered with coarse cloth such as salt bags. The bottom of the filter communicates with the bottom of a conveniently located cylinder from the top of which connection is made with a suction-pump. After all the salt has been transferred to the filter the suction-pump is started and connection on the filter with the cylinder in which a partial vacuum has been produced is made, whereupon the adhering half-crude is transferred to the cylinder together with what wash-water it may be necessary to use. Further purification of the salt may be effected by placing wooden covers on the filter and blowing live steam over the salt, partial vacuum meanwhile being maintained in the cylinder. After sufficient purification the dry salt is removed to be used again for graining soap. The wash-liquor from it is transferred to the half-crude storage-tank to be evaporated as required. The half-crude, as stated, is concentrated to crude which naturally is obtained in reduced volume.

Utilization of Recovered Salt.—The salt as removed from the filter is of varying degrees of purity according to the thoroughness of the purification of the waste lye, whether hydrochloric or sulphuric acid has been used as the neutralizing agent, whether a portion of the crude sulphate has been separated by fractional crystallization, previously described, and according to the thoroughness of the washing process. The impurities may be divided into two classes, viz., organic and inorganic. The former comprise fatty, resinous and albuminous matter imperfectly removed from the waste lye and crude glycerin remaining after the washing process in the filter. The latter may compromise sodium hydrate and carbonate, incompletely neutralized in the preliminary purification of the waste lye and adhering in solution to the salt and sodium sulphate resulting from the use of sulphuric acid in neutralization, and oxide of iron from the interior of the evaporator, or iron salts not completely removed in the purification of the waste lye. In addition there is more or less water. The salt may be used directly in the kettle without further treatment. When dark it is not without discoloring effect upon the soap which, while not affecting rosined soap to an appreciable degree prevents its use for graining white soap. It may be purified, when desirable and practicable to use it in saturated solution, by dissolving it in water; treating it with sufficient diluted sulphuric acid to aid removal of the organic matter by filtration, and treating the filtrate thus purified with soda-ash to precipitate the iron salts, whereupon it is again filtered and is ready to be used.

When unpurified, organic impurities pass again into circulation, which should be prevented whenever practicable by using the inferior recovered salt on nigres. With proper purification of waste lye the salt recovered is of bright color and suitable for use on any change. With varying proportions of sodium sulphate present its graining efficiency is correspondingly reduced.

Crude Glcyerin.—Crude glycerin is a saturated solution of various alkaline salts in glycerin together with varying amounts of organic impurities. The commercial value of crude glycerin depends upon its content of glycerin, which in turn in crude glycerins of the same density is affected by the proportion and character of the impurities. These vary largely with the source of the crude glycerin, which in the order of the general quality of the product may be stated as follows: Candle-stock saponified by steam under pressure; waste soap lye; and candle-stock subjected to acid saponification and distillation. The first and third sources and their respective products already have received attention.

The proportion and amount of impurities of crude glycerin obtained from waste soap lye are determined by the degree of the purification of the waste lye and the materials used therefor, and the degree of concentration of the product. The impurities eliminated during concentration are in general inorganic and remain to constitute about 10 per cent of the weight of the concentrated product. As concentration proceeds the proportion of organic matter, imperfectly removed by the initial purification of the waste lye, increases and affects the color, body and commercial value of the crude glycerin. A high proportion of organic impurity not only means a reduced yield of the refined product but its more or less deterioration and increased difficulty in obtaining the yield. The proportion of common salt increases as the salting-point is reached, whence it falls off to the average percentage occurring in good crude glycerin, viz., about 8 to 10 per cent. Alkaline salts increase in proportion as concentration proceeds owing to their greater solubility. The color of crude glycerin depends upon the proportion of foreign organic matter present, and this is determined not so much by its source as the thoroughness of purification of the original aqueous solution, whether it be sweet water or waste soap lye.

As is clear the proportion and amount of the various saline constituents will be very materially affected by the source, being less with sweet water than with waste lye and the variety depending upon the procedure employed.

The following average analysis of crude glycerin made by Richardson and Jaffé is typical:

	Per Cent.	
Glycerin.	77.30	
Fat acids (as soap)	0.80	
Soda (Na_2O) (as soap)	0.08	10,21 per cent of "salts."
Sodium carbonate	2.80	
" chloride	6.56	
" sulphate	0.34	
Alumina and silica	0.43	
Water (with traces of organic coloring matters)	11.69	
	100.00	

Distillation of Crude Glycerin.—Various forms of distilling apparatus have been devised; some of them impracticable and some of them used until supplanted by a more efficient form. They comprise essentially a vessel for containing and heating the crude glycerin, a device inserted therein for the introduction of agents to facilitate distillation, and suitable apparatus for the condensation of the distillate. The form of distilling apparatus at present in general use, like every complete machine, contains in its structure vestigial characteristics of every mechanism of any degree of practicability that along the same lines has preceded it.

The system of glycerin distillation covered by the patents of Jobbins and Van Ruymbeke has as its distinctive characteristic the introduction into the distilling vessel wherein high vacuum is maintained and into the mass of crude glycerin therein contained, a jet of expanded and reheated steam. The system comprises a suitable vessel for the expansion and reheating of high-pressure steam, a distilling vessel, suitable receptacles for the distillate and an air-pump for the maintenance of a high degree of vacuum.

Construction of Reheater and Still.—Referring to Fig. 213, 66 is a reheater made preferably cylindrical in shape, of iron or of steel, and 67 is a still. The heater 66 and still 67 are vertically arranged side by side, as shown. 68, 68, 68 are pillars or posts having the braces or I beams 71 at the top thereof. The heater 66 and still 67 are supported by these braces by means of lugs or brackets 97 bolted to the same. 72 is a steam-pipe from a boiler or any suitable source of steam-supply. This pipe 72 connects with the steam-pipe running parallel midway between the heater and the still and provided at its lower end with the enlarged cylindrical chamber 74, for receiving the water of condensation from the steam-pipe. Beneath this chamber 74 is a valve 75, for removing the water of condensation. 76 is a small steam-pipe connected with the pipe 73 above the chamber 74 and provided with a valve 77 therein. The steam-pipe 76 enters the heater 66 near the lower part thereof and connects with the steam-coil 78 in the heater. This steam-coil 78 is preferably made of considerably larger diameter than the steam-pipe 76, so that the steam from the pipe 76, on entering the coil 78, will rapidly expand therein, its temperature being correspondingly reduced. The steam-coil 78 is supported within the heater 66 by means of braces 78′ arranged vertically in the heater. The upper end of the coil 78, which is called the "expansion coil," connects with the pipe 80, which passes out through the side of the heater and enters the still 67, being provided at its lower end with a distributing ring or pipe 81, having perforations therein preferably extending downward for injecting steam into the contents of the still. 82 is a pipe extending out from the pipe 72, and provided with a valve 83 therein, said pipe 82 entering the top of the heater 66. 84 is a pipe at the bottom of the heater 66, provided with a valve 85 therein, for removing water of condensation from the heater. 86 is an asbestos or other insulated covering around the heater 66, in order that the heat of the same may be retained without loss by radiation. 87 is a draw-off pipe provided with a valve for removing the contents of the still when desired. 89 is a door in the lower part of the still, provided with a valved pipe 90 for removing the sediment and residue from the still. 91 is a water-gage or column on the still 67 for determining the amount of material therein. 92 is a pipe branching out from the steam-pipe 72 and having a valve 93 therein. This pipe 92 connects with a steam-coil 94 within the still for heating the contents thereof. 88 is a valved steam pipe connected with the lower end of the coil 94 for carrying off the steam or condensed water therefrom. 95 is a pipe having valves 96, for introducing the material into the still 67. 98 is a pipe for carrying off the distillate or vapor from the still 67, which pipe connects with the upper end of the drum 69. 100 is a pipe connecting the upper

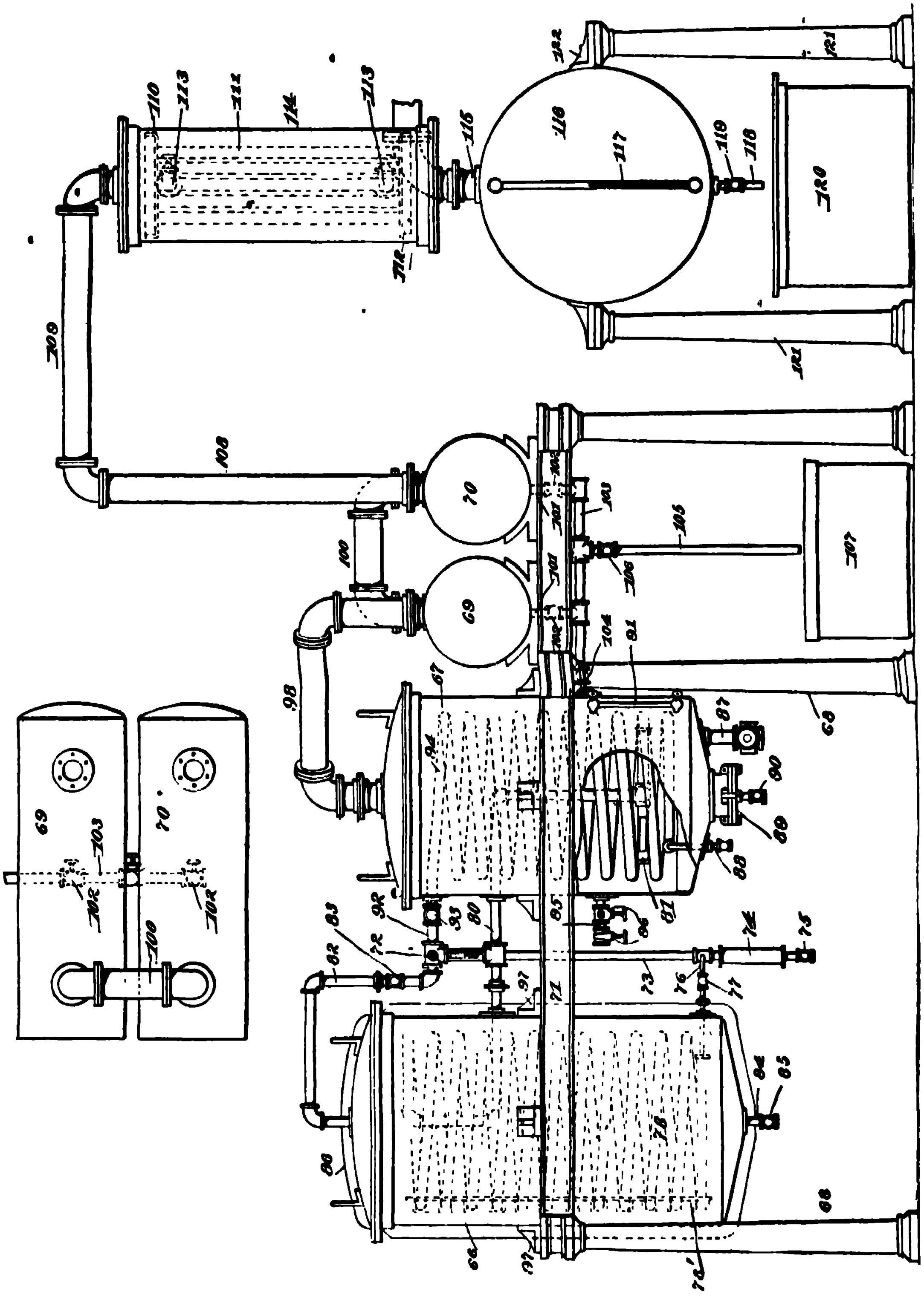


FIG. 213.—Jobbins Distilling Apparatus.

end of the drum 69 with the upper end of a similar drum 70 arranged at the side of the drum 69, both of these drums being supported on the braces 71 by means of the saddles or brackets 99. 101 are pipes leading out from the bottom of the catch-all drums 69 and 70 and provided with valves 102 therein. The pipes 101 connect at their lower ends with the pipe 103 which enters the side of the still and which is provided with a valve 104. 105 is a pipe connected with the pipe 103, which enters the side of the still and which is provided with a valve 106 therein. 107 is a tank beneath the pipe 105. Instead of making use of two drums, as described, it is obvious that only one drum, or three or more drums, may be employed. Extending up from the top of the drum 70 is a vertical pipe 108, provided with a horizontal branch pipe 109 at its upper end. This pipe 109 connects with the upper end of the upright cylindrical condenser 114. Within this condenser are the heads or drums 110 and 112 connected together by tubular flues 111. 113 are pipes leading into the top and out of the bottom of the space formed between the heads 110 and 112, for maintaining the circulation of water or other cooling agent therein. 115 is a pipe leading out of the lower end of the condenser 114 and connected with the receiving drum 116. This receiving drum 116 is provided with a water-gage 117 thereon, for determining the amount of condensed distillate therein. The said drum is also provided with a draw-off pipe 118, having a valve 119 for drawing off the condensed distillate. A tank 120 placed beneath the receiving drum 116 may be used for receiving this distillate. The receiving drum 116, and also the condenser 114 are supported on pillars or posts 121 by means of lugs or brackets 122, bolted to the tops of the pillars and to the side of the drums.

Operation of Still.—A sufficient quantity of crude glycerin is introduced into the still 67, Fig. 213, through the pipe 95 preferably by reason of the vacuum therein. The valve 96 is now closed and vacuum maintained in the still. Steam is introduced through the pipe 72 and, passing through the pipe 92 into the steam-coil 94, heats the contents of the still up to a temperature approximating the boiling-point thereof at the vacuum used. Steam from the pipe 72 also passes through the pipe 82 into the heater 66 and heats the expansion-coil 78 therein to the proper temperature. Steam from the pipe 72 also passes through the pipe 82 into the heater 66 and heats the expansion-coil 78 therein to the proper temperature. Steam from the pipe 72 also passes through the pipe 73 into the smaller pipe 76, and thence into the large expansion-coil 78 where it rapidly expands, its temperature being thereby reduced corresponding to the degree of expansion. By heating the expansion-coil 78 by means of free steam in the heater 66, the expanded steam in this expansion-coil will be reheated approximately to its original temperature, and in its expanded and reheated form will pass through the pipe 80 and be injected into the material through the perforated pipe or ring 81. By first allowing this steam to expand and by then reheating it before injecting it into the liquid, expansion in the still in the presence of the vacuum used is reduced to a minimum. This injected steam, at a temperature of about 300° Fahr. at a vacuum of 28 inches or more, in passing through the liquid will rapidly distill the same, and the distillate passes out through the pipe 98 into the drum 69. The less volatile distillate, comprising glycerin, generally of an excellent quality, will be deposited in the drum 69. From the drum 69 the distillate passes by means of the pipe 100 into the drum 70, and more glycerin will be condensed and retained therein. The more volatile distillate, consisting of glycerin with a large percentage of watery vapor, passes up from the drum 70 by means of the pipe 108 and enters the condenser 114 where it is condensed by passing through the cooling pipes 111. The condensed distillate will now be deposited in the receiving drum 116. The sweet-water thus collected in the drum 116 may be removed therefrom into the tank 120 after the distillation has ceased, or by stopping the vacuum-pump. The less volatile distillate collected in the drums 69 and 70, which as before mentioned, is concentrated glycerin, generally of a bright color and of an excellent quality, may be drawn out of said tanks into the tank 107 through the pipes 101, 103, and 105, but if upon test it is found that this glycerin is not of sufficient purity, it may be returned through the pipe 103 into the still by opening the valve 104.

On starting the still it is first necessary to discharge any water that may have been allowed to remain in it for cleaning. The reheater is then heated up slowly and the vacuum-pump started.

Crude glycerin is then drawn in the still by aid of the vacuum up to the established level for safe operation and at which it is maintained without great alteration until near the end of the distilling period, when it may be allowed to subside from the distillation of the last portions of glycerin advisable from the accumulated foots. After introducing the requisite amount of crude glycerin the still and contents are heated, whereupon the jet of expanded and reheated steam may be applied slowly and with great care to avoid entrainment of matter into the catch alls.

In shutting down the still it is first necessary to close the jet, to close the steam-valve admitting steam to the heating-coils of the still and heater, to turn off the cooling water to the vacuum-pump and to stop the pump. Vacuum on the still is now gently broken through the jet-pipe sufficiently to clean the latter, whereupon the vacuum break is opened. The distillate is now discharged separately from the catch-all and sweet-water drums, and finally the foots, into tanks placed beneath the drums and the still. Water is now introduced into the still which is heated to boiling, to clean the interior, by means of steam through the jet-pipe, after which the wash-water is discharged into the foots. Where the vacuum in two stills is maintained by the same pump it is necessary in shutting down one still to close all connections of that one with the other.

Expanded and Reheated Steam. — The particular advantage which properly expanded and reheated steam possesses in distillation is, that the steam expands but slightly and consequently causes but slight absorption of heat due to such expansion. When on the other hand, ordinary free steam is used for distillation it always expands very much in the still, and absorbs heat from the contents of the still, so that distillation is retarded not only because of the diminution in temperature thus produced in the material, but also because of the varying quantities of condensed steam retained by the material.

With superheated steam, that is to say, steam which has been heated to a high temperature by fire-heat or by other means, the same objections are found, and although the diminution in temperature suffered by super-heated steam in expanding in the still, whether a vacuum is used or not, may not be sufficient to cause condensation, yet such steam is undesirable for the distillation of glycerin or similar materials, since decomposition products will be inevitably formed.

Although the expansion of the steam injected may be varied within a very wide limit, it has been found that steam coming from the boiler at a pressure of from 70 to 120 pounds, when expanded to from twenty to thirty times its original volume, will give excellent results. Although a slight expansion and reheating of the steam to be injected is beneficial *pro tanto*, still it is preferable that the injected steam be expanded until it has a pressure less than that of saturated steam at the boiling-point of the liquid from which the glycerin is distilled. To obtain the best results it is also necessary that the temperature of the injected steam be kept strictly below the point at which decomposition products are formed. When a high vacuum is used, the pressure of the injected steam may be less than atmospheric pressure.

Products of Distillation.—The products of distillation are three in number, viz., heavier portions of the distillate called "half-refined" glycerin, collected in the catch-alls 69 and 70, "sweet-water" (to be distinguished from the sweet-water of steam or acid saponification), collected in the sweet-water drum 116, and "foots" or the residue left in the still on the termination of the distilling period. This residue is of dark color and of tarry consistency and which, on account of the large amount of sodium acetate and other organic soda salts therein contained, can no longer be distilled for glycerin, yet as the glycerin in such "foots" is unaltered it can be recovered and transformed into crude glycerin by a process to be described later.

The half-refined glycerin is subjected to a second distillation, commonly in a second distilling-plant reserved for that purpose and for the distillation of the concentrated sweet-water. This course is made necessary because it is impossible to completely prevent small quantities of crude glycerin from being entrained or carried off with the distillate, which after concentration will often contain one-hundredth of one per cent of sodium chloride. The glycerin is also generally apt to be contaminated with a slight quantity of an organic impurity of a resinous nature, which, as it has a lower boiling-point than the glycerin, comes off with the first portion of the distillate, and may thus be separated either from the first or from the second distillate.

Construction of Concentrators.—Referring to Fig. 214, 123 represents a concentrator made preferably either of iron or steel. This concentrator 123 is supported on pillars or posts 124 by means of brackets or lugs 125 bolted to the concentrator, and to braces 126 connecting the tops of the posts. 127 is a steam-pipe provided with a valve 128, having a steam-gage thereon for registering the amount of pressure in the pipe. This pipe connects with a steam-coil 129 within the still for heating the contents thereof. 130 is a steam-pipe having a valve therein for carrying off the condensed steam from the coil 129. 131 is a water-gage or column on the side or front of the concentrator 123 for indicating the height of the material therein. 132 is a vacuum draw-off pipe connected with the concentrator 123 at one or more levels, and provided with valves 133, 134 for drawing material into pipe 132 from any desired portion of the liquid in the concentrator. 135 is a valve beneath the pipe 132 for removing the material drawn therein for the purpose of testing the same. 135′ is a pet-cock at the top of the pipe 132 for admitting air into the same. 136 is a draw-off pipe at the lower end of the concentrator for removing the concentrated material therefrom. 157 is an annular dash-plate within the concentrator near the top of the same, and provided with a circular dash-plate 138, supported beneath the plate 137 by means of rods 139. 140 is the opening in the annular plate 137 above the circular dash plate 138, but of smaller diameter than the same. 141 is a pipe from the concentrator 123 for carrying off the aqueous vapors and other volatile matters therefrom, and provided with a valve 142. This pipe 141 is connected with a catch-all 143 provided with a pipe 144 therein, having its open upper end arranged above the opening of the pipe 141. 145 is a water-gage for indicating the amount of liquid that may be caught by and retained in the catch-all 143. 146 represents the continuation of the pipe 144 and which connects with the top of the drum 147, provided at one of its ends with a water-gage 148. 149 is a draw-off pipe for removing the contents of the drum 147, provided with a valve 150. 151, 151 are pillars or posts on which the drum 147 is supported by means of brackets 152 bolted to the top of said pillars or posts and to said drum. 153 is a pipe which leads out from the bottom of the catch-all 143 at one side of the pipe 144, and is connected with a horizontal pipe 154 by means of a four-way casting, as shown, to enable the pipes 153 and 154 to be cleaned. The pipe 154 enters the side of the concentrator 123 and is provided with a valve 154′ thereon. 155 is a valved pipe entering the pipe 154 between the concentrator and the valve 154′ in said pipe, for introducing the material into the concentrator. 156 is a second concentrator provided with a steam-pipe 157 having a valve 158 therein, and a steam-gage 159 thereon. This steam pipe 157 connects with a coil 160 on the inside of the concentrator 156. 161 is a water-gage on the concentrator, and 162 is a vacuum draw-off pipe connected therewith and having valves 163, 163. 164 is a draw-off pipe for the concentrator and having the valve 165, and 166 is a steam-pipe connected with the end of the coil 160 for carrying off the steam therefrom, or for removing the water of condensation therein. 167 is a pipe having valves 168 for introducing material into the concentrator 156. 169 is an annular dash-plate near the top of the concentrator 156 provided with a circular dash plate 170, supported by rods 171, as before explained. 172 are pillars or posts for supporting the concentrator 156 by means of lugs or brackets 174 bolted to the braces 173 and to the concentrator. 175 is a pipe for carrying off the aqueous vapors from the concentrator 156, and provided with a valve 176, which pipe 175 connects with the catch-all 143. In practice it is desirable that the concentrator 156 which concentrates the glycerin after the second distillation should be made of copper preferably tinned on its interior, in order that there may be no danger of contaminating or discoloring the glycerin.

Concentration and Utilization of Sweet-water.—Referring to Fig. 213, the distillate in the tank 120 is now ready for the first concentration in the concentrator 123, Fig. 214, which may be an iron concentrator. The valve 176 is closed so as to cut off the concentrator 156 and a vacuum is maintained in the concentrator 123 by means of a vacuum-pump connected with the receiving drum 147. By thus maintaining a vacuum in the concentrator, the glycerin from the receiving tank 120 may be drawn through the pipe 155 by suction, or by any other suitably located pipe. Steam is now admitted into the coil 129 through the pipe 127 and the glycerin is maintained at a temperature above the boiling-point of water at the vacuum used. The watery vapor thus driven off passes through the pipe 141 and enters the catch-all drum 143. This watery vapor generally carries with it small and varying quantities of glycerin. The watery vapor and glycerin passing into the catch-all drum will impinge against the pipe 144 and the glycerin contained in the vapor will condense and deposit in the drum 143. The watery vapor thus driven off passes into the pipe 144 and accumulates in the receiving drum 147, from which it may be removed as desired.

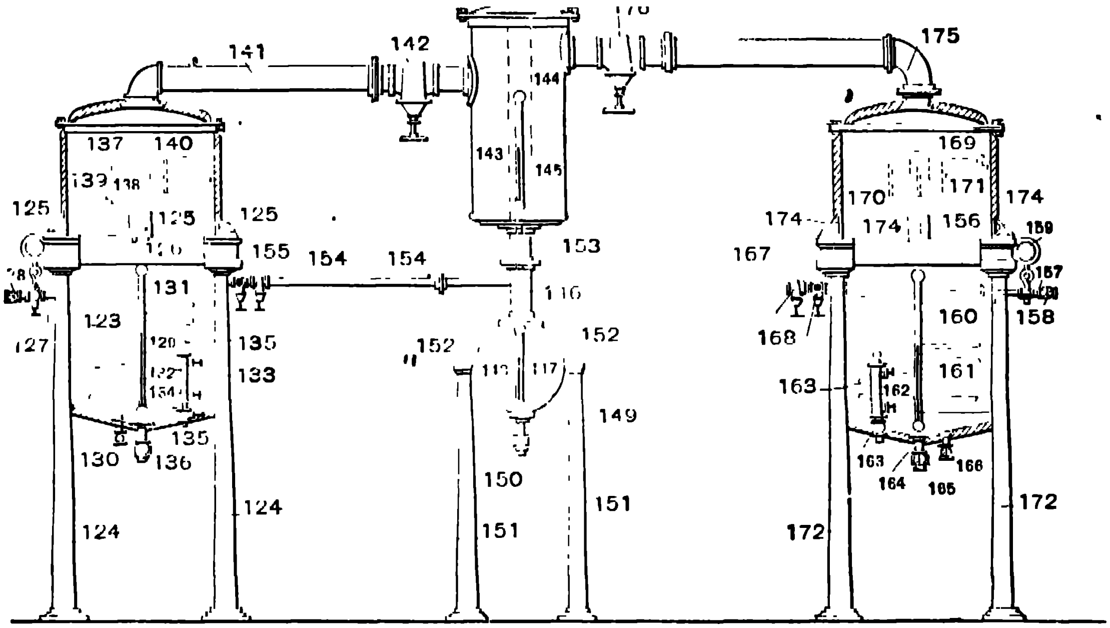


FIG. 214.—Sweet-water and Glycerin Concentrators.

The glycerin which is condensed in and collected by the catch-all drum 143, by reason of the fact that it is of a less volatile nature than the watery vapor passing through the same, may be returned to the concentrator 143 by the pipe 154 by opening the valve 154′ therein. When the glycerin has been sufficiently concentrated, which can be determined by means of the pipe 132, it is passed through another distilling apparatus, preferably like that illustrated in Fig. 213 except that it is advisable that the catch-all drums 69 and 70, condenser 114, and receiving-tank 116 of the second still should be made of copper lined with tin, so as not to contaminate the glycerin of the second distillation.

Concentration of Refined Glycerin.—The heavier portions of the distillate, referred to as half-refined glycerin, obtained from the distillation of crude glycerin, as already described, are subjected to a second distillation in a distilling apparatus identical in construction and operation to that used and described for the distillation of crude glycerin. The products of this distillation are refined glycerin, residual foots and sweet-water, collected respectively in the catch-alls, still, and sweet-water drums. The sweet-water is concentrated, as already described, the concentrated product being distilled either separately, which is preferable, or in combinatian with half-refined glycerin in the still reserved for the distillation of half-refined glycerin. To combine with the volatile fatty acids, which may be present in varying amount, and thus to a degree to retain them as alkaline salts in the residual foots, soda-ash is usually added to the tank containing the concentrated sweet-water and well mixed through prior to distillation. The heavier portions of the distillate obtained from the distillation of half-refined glycerin constitute refined glycerin, which undergoes a final concentration in concentrator 156, Fig. 214.

After this second distillation the glycerin is introduced into the concentrator 156 through the pipe 167 and is heated so as to drive off the water therein by steam in the steam-coil 160. The watery vapor passing out of the still 156 enters the catch-all 143 by means of the pipe 175, and is condensed and collected in the receiving drum 147. This concentration is carried on until the distillate has reached a specific gravity of 1.262 for dynamite purposes, or even higher if necessary.

It is obvious that by making use of a catch-all 143 and in providing means for returning the glycerin caught therein back to the concentrators, the loss of glycerin during the operation of concentration is reduced to a minimum.

Clarification of Refined Glycerin.—The tendency during the concentration of refined glycerin is for it to darken, which tendency is greater in proportion to the water it contains. The darkening, however, is to be attributed not so much to the presence of water alone as to the presence of non-glycerin organic matter accompanying the water. After the desired specific gravity has been obtained by operating the concentrator, as has been described, the condensing water is shut off, the pump shut down, vacuum in the concentrator broken and the contents discharged through the draw-off pipe 164, into a tank of suitable capacity placed beneath the concentrator. Ordinarily the glycerin is discharged directly from the concentrator into drums, but where conditions warrant the treatment, a small amount of boneblack may be added to the hot refined glycerin and the whole thoroughly mixed by stirring, the utmost cleanliness being observed meanwhile. The mixture is then filter-pressed in a press reserved for this sole purpose and the clarified filtrate transferred to a storage-tank for shipment as required. This product is of a pale-straw color and comes on the market as dynamite-refined glycerin, so-called from its chief use, viz., the manufacture of nitroglycerin which, in admixture with infusorial earth to impart body, is known to engineering and the arts as dynamite. Colorless water-white glycerin of the pharmacopœia is produced by subsequent distillation and clarification of the dynamite refined grade under conditions wherein all tendency to discoloration is eliminated.

Utilization of Glycerin Foots.—In the processes of distilling glycerin generally employed when the distillation has reached the point where the glycerin ceases to come over there is left in the still a tarry residue resulting from the decomposition of the glycerin, and of organic matter accompanying the glycerin, which decomposition entails a loss varying from 5 to 12 per cent of the glycerin contained in the waste lye.

This residue, commonly called glycerin foots, contains a large percentage of glycerin (33 per cent and over), but which cannot be distilled off in the ordinary way. Glycerin foots contains, in addition to the glycerin, generally about 5 per cent of salt, from 20 to 30 per cent of acetate of sodium, and from 10 to 15 per cent of sodium salts of non-volatile organic acids, together with some minor impurities.

Glycerin is recovered from glycerin foots by transforming the sodium salts of organic acids into sodium salts of inorganic acids by treatment with mineral acid, commonly sulphuric acid. The procedure of recovery is based on the fact that compounds of soda and a mineral acid, such as sodium sulphate or chloride, are much more readily separated from soap lye, chemically purified and boiled down to salting-point, than the organic sodium salts which may be decomposed by these mineral acids.

The procedure of recovery is substantially as follows: The percentage of sodium carbonate which the glycerin foots will yield by combustion, which can be determined by incinerating a small portion of the glycerin foots, having been ascertained, the amount of sulphuric acid which will be necessary to neutralize the sodium salt in the glycerin foots is calculated. This quantity of sulphuric acid is added to the foots, together with wash-water from the still, which meanwhile have been transferred by pump from the drop-tank beneath the still to a suitably located lead-lined tank. On the addition of the sulphuric acid to the mixed foots and water and with thorough agitation the sodium compounds of organic and inorganic acids therein are immediately converted into sodium sulphate. During the reaction much heat is developed and the organic matter is more or less carbonized. After standing a short time the mixture is allowed to run by gravity through coarse bag-filters, or it may be filter-pressed. The filtrate is then subjected to heat in a specially constructed cast-iron still, whereupon the volatile organic acids, chiefly acetic, are expelled. Evaporation may be continued as far as desired after this stage. The product, as a rule, is then transferred to the waste lye mixing-tank, the glycerin contained therein being added to that of the waste lye, and the whole purified by the usual treatment with sulphuric acid and persulphate of iron.

In the practical operation of the crude-glycerin still it is found that it is not convenient to continue the distillation of the glycerin beyond the point where the glycerin foots contain from 50 to 60 per cent of glycerin, because at this concentration the glycerin foots are sufficiently fluid to be readily removed from the still, whereas if the concentration is carried further the mass becomes too viscid, and if the distillation be carried to the point where the glycerin foots contain only 30 per cent of glycerin the mass becomes quite solid when cold. Besides in this last concentration the glycerin distils off very slowly, causing loss of heat.

Garrigues Process of Glycerin Recovery. — By a combination in sequence of various common chemical reactions, Garrigues has devised a process of treating waste lye preliminary to and during concentration whereby the volatile fatty acids are claimed to a large degree to be eliminated previous to distillation. In the processes hereinbefore described volatile fatty acids either escaped in the water of condensation used in concentration, or collected in the sweet-water condensed in the cooling towers of the distilling system which sweet-water was later concentrated and neutralized with soda-ash previous to distilling, or were set free from their alkaline salt by treatment with sulphuric acid in the particular treatment to which the glycerin "foots" was subjected. In our study of this process we will confine our attention first to its theoretical consideration, and will then discuss in detail the construction and operation of the apparatus used. To learn what improvement is possible over similar and prior processes this process should be studied in connection with the description of the former.

First. Neutralization of the free and combined alkalinity of the waste lye is effected by means of sulphuric acid. The insoluble fatty acids before in solution as soap are hereby also set free. By the use of common alum as a clarifying agent insoluble aluminum soap is precipitated. The waste lye thus treated under suitable conditions is then filtered. The soluble fatty acids remain in solution and pass with the filtrate which is now concentrated until the residual liquor contains from 25 to 40 per cent of glycerin. In this operation about eight-tenths of the chloride and sulphate of sodium crystallize out and may be separated in the usual way.

Second. To the half-crude glycerin barium chloride is now added with the result that the sodium sulphate remaining in the half-crude is precipitated as insoluble barium sulphate. In addition any soapy matter still present is decomposed and precipitated as an insoluble barium soap. Now sufficient sulphuric acid is added to decompose the greater part or all of the alkali compounds of the volatile fatty acids whereby the latter are set free and in turn are precipitated as insoluble barium compounds. The mixture is then filtered. Sulphuric acid is added only after the barium sulphate has been produced in the liquid and not in such excess as to decompose the barium soap already precipitated.

Third. The half-crude glycerin thus treated is now reduced by evaporation to crude glycerin containing from 70 to 90 per cent of glycerin, whereby a more complete separation of salts is effected, together with the elimination of the greater part of the volatile fatty acids in the condensing water.

The term "volatile fatty acids" as here applied is used among chemists to mean such fatty acids as vaporize with water vapors at a temperature not exceeding 212° Fahr. These include acetic, butyric, caprylic, etc., acids. As the glycerin becomes more concentrated in the remaining liquid by the evaporation, a combination takes place between some glycerin and part of the remaining volatile fatty acids. Hereby the glycerides of those acids are produced which are presumably identical with the glycerides contained in the original fats, from which latter they were extracted during the process of making soap. Therefore, it is not feasible to eliminate all the volatile fatty acids present during one evaporation.

Fourth. The next step in the process is to distil off the glycerin. At the high temperature necessary for doing this the glycerides of the volatile fatty acids contained in the liquid would be again decomposed into free fatty acids and glycerin. Both would distil, and some glycerides would also distil unchanged. To avoid contamination of the distillate with the acids or glycerides, both of which are objectionable, sufficient soda-ash is added to the contents of the still. This combines with all the fatty acids present no matter whether they exist in the free state or in combination as glycerides. Now the liquid is submitted to distillation, the glycerin passing over and the fatty acids being retained in the still with some remaining salts. This is the case because the fatty acids are bound by the alkali and are now probably in their original condition in which they existed in the lye. The distillation is continued until practically all the glycerin has been distilled, the residuum in the still consisting, essentially, of sulphate and chloride of sodium, the alkaline salts of the fatty acids just mentioned, and some coloring-matter from the fats, together with a small quantity of glycerin. This residue is so small that it may be discarded, or, if deemed advisable to save the salts and the small amount of glycerin, it may be added to a fresh supply of waste lye and again put through the process.

The insoluble fatty acids combined with alkali and in solution as alkali salts instead of being separated out by the use of sulphate of aluminum may also be rendered insoluble by the addition of sufficient mineral acid, whereby said insoluble acids will be set free. This would save the trouble and expense of forming aluminum soap; but unfortunately the free fatty acids present a mechanical difficulty during filtration, because they are sticky and soon clog the pores of the filtering medium. For this reason the formation of an insoluble soap is preferable, because it permits of quick filtration.

Theoretically considered the elimination of volatile fatty acids may be brought about if in the first treatment the waste lye is made acid to such an extent that the volatile fatty acids are set free instead of doing this later in the partly-concentrated liquid. These would then pass over with the water-vapor during the subsequent evaporation of the liquid. This plan is open to physical objection, because the dilute liquid will froth violently during the evaporation if it contain free volatile fatty acids. However this does not take place if the acid condition of the liquid is produced after the same has become more viscid due to a greater percentage of glycerin therein after evaporating. Furthermore, the bulk of dilute liquid to be handled would be so great that it would not be easy to get the required conditions which are easily obtained when a small quantity of more concentrated liquid is to be treated. In addition thereto the waste lyes contain some fatty acids which are sparingly soluble in water, but not readily volatile with water-vapor. If the dilute liquid is acidified, then these fatty acids pass into the filtrate by virtue of the relatively large amount of water present in the dilute lye. If, on the contrary, a small bulk of concentrated liquid is acidified, then these acids are largely precipitated and removed in the second filtration. The barium chloride above mentioned is mainly added for the purpose of enveloping these fatty acids with sulphate of barium. The free fatty acids are less soluble in water than their barium salts.

The distinctive features of this process of glycerin recovery may be summarized as follows: There is in the still a very small amount of volatile fatty acids in comparison to the quantity of glycerin present. As these volatile fatty acids are partly distilled with the glycerin even from an alkaline liquid, and as the larger the quantity in the still the more of them are distilled, it is apparent that this process greatly reduces the amount of fatty acids found in the distilled glycerin. In addition to this advantage it is claimed that the process further increases the distilling capacity of a given distilling apparatus. The rate of distillation is materially retarded by the presence of the alkali salts of the volatile fatty acids in the liquid to be distilled. Furthermore, it is impossible to completely separate the glycerin from the alkali salts of the volatile fatty acids. Some of the fatty acids pass over into the distillate, and, on the other hand, some glycerin is retained by the salts in the residue. The glycerin thus retained increases in mathematical proportion to the quantity of the salts of the volatile fatty acids in the still. By eliminating the volatile fatty acids almost entirely in this process previous to distilling the glycerin there is produced a residue so low in volatile fatty-acid salts that the glycerin retained by them is trifling, and the residue may be economically discarded. The residues resulting from methods formerly practiced contain so much glycerin that they cannot be economically discarded and the glycerin has to be recovered therefrom by mixing it with fresh raw material. In some instances the residues contain 50 per cent of glycerin, which could not be distilled off conveniently, while by this process of treatment it is possible to distil off practically all the glycerin. There are also in the market residues from the evaporation of soap lye, from which the insoluble fatty acids, chlorides, and sulphates have been largely separated. These may likewise be treated by this process. In this case water is first added to the liquid, and the diluted liquid thus obtained is then treated with acid or chloride of barium and acid. The precipitate formed thereby is separated from the remaining liquid, the water and volatile acids evaporated, the residue made alkaline, as above described, and the glycerin distilled off.

Operation of Crude Glycerin Plant, Garrigues System.—The practical operations involved in the preparation of waste lye for

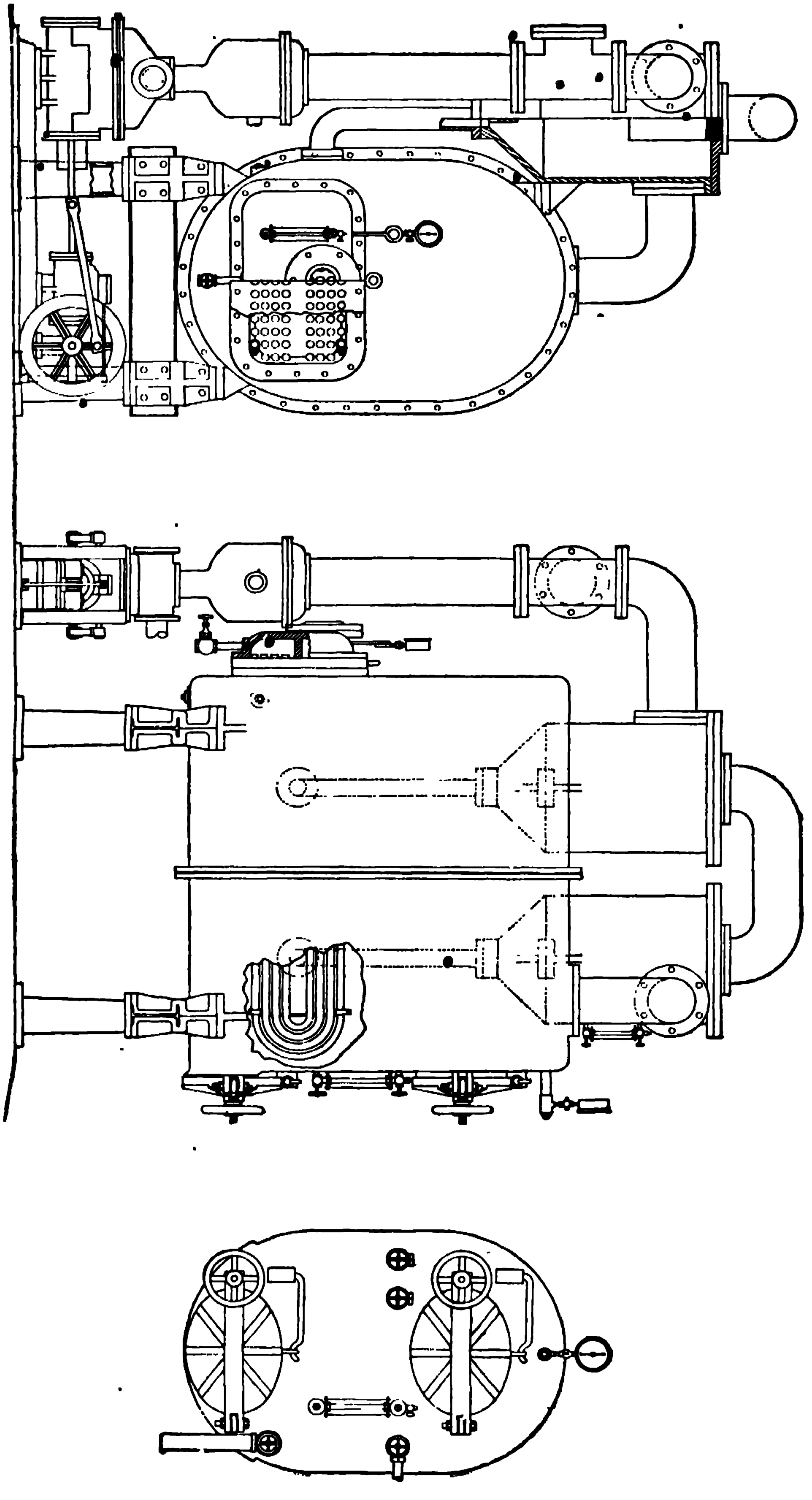


FIG. 215.—Garrigues Evaporator, Single-effect.

evaporation by the Garrigues process, the theory of which has been dwelt upon previously, may be followed by referring to Fig. 216, in which is shown a complete plant for the production of crude glycerin.

To the waste lye in the treatment-tank, to which the waste lye in measured amount has been transferred from the storage-tank, is added about one-half of one per cent of sulphate of alumina, or common alum, and sufficient sulphuric acid to nearly neutralize the caustic soda and sodium carbonate present Agitate the contents of the treatment-tank by means of air introduced therein through a perforated coil (not shown) from the pipe 24, Fig. 216. Injection of air is effected by means of a Koerting exhauster, valve 7 being closed and valve 15 opened. After sufficient agitation of the treated lye, filter it into the feed-tank by starting the pump, previously opening valves 20, 18, and 21.

With a supply of clear lye ready for evaporation, the operation of the evaporator may now be followed: Exhaust the air from the evaporator by opening valve 2, Fig. 216, admitting water to the vacuum-pump and then starting the vacuum-pump. Admit clear lye from the feed tank into the evaporator by opening valve 1, allowing the evaporator to fill to just above the level of the tubes, after which close valve 1. Now admit steam into the tubes of the evaporator, and when boiling begins, again open valve 1 just enough to admit clear lye in sufficient volume to replace the decrease in volume by evaporation, maintaining thereby a constant level in gauge-glass 31. Continue to feed clear lye in this manner until the concentrated liquor begins to appear heavy in the gauge-glass, at which stage it will contain about 40 per cent of glycerin. This requires about four hours, maintaining a steam-pressure of one pound in the tubes and a vacuum of 25–27 inches in the evaporator.

When a satisfactory density of the concentrated liquor has been obtained, shut off steam to the tubes and close valves 1 and 2; stop the vacuum-pump and open valve 13, which breaks the vacuum. Open valve 3 and allow the hot concentrated liquor to flow into the salt-filter. Now open valves 4, 5, and 6 and pump the liquor to the settling-tank. When the liquor has drained from the evaporator, open the salt-door and transfer the separated salt from the space beneath the tubes to the salt-filter. Draw the liquor from the salt in the filter to the settling-tank by means of the pump and finally start the Koerting exhauster (valve 15 being closed, valve 7 opened, and valve 8 partly opened). By this means a vacuum of about 20 inches is maintained beneath the body of salt in the filter, whereby the adhering liquor effectually is removed and the salt dried. The air and liquor separate in the stand pipe, the liquor passing to the pump and the air to the outside through valve 7. Any froth that passes the stand-pipe is separated in the steam-separator and returns to the treatment-tank through valve 8.

When the concentrated liquor, containing as stated about 40 per cent of glycerin, has collected in sufficient volume in the settling-tank it is transferred to the treatment-tank and about one-half of one per cent of barium chloride is added, whereupon is formed a precipitate consisting of barium soap, barium chloride, and barium sulphate. A filtered sample of the concentrated liquor, which may be called "half crude," is now tested for volatile fatty acids by the following method: To 10 grams of the sample are added 140 cubic centimeters of distilled water and one cubic centimeter of concentrated sulphuric acid. Connect the flask with a Liebig's condenser and distil off 100 cubic centimeters. Titrate this volume with standard caustic-soda solution using phenolphthalein as indicator. Calculate the organic acidity in terms of sulphuric acid, and add the necessary quantity of sulphuric acid to the charge of half-crude in the treatment-tank. It may be added that this method does not measure the entire amount of volatile fatty acids, but the discrepancy is immaterial, for it is always on the safe side, thus avoiding an excess of free sulphuric acid in the half-crude. After thorough agitation of the charge of half-crude, proceeding as already explained in the treatment of waste lye, filter the charge into the feed-tank. Fill the evaporator, as described before, with half-crude, maintaining, however, with this material a steam-pressure in the tubes of about 20 pounds. During the second stage of the evaporation, which is carried on until the level of the liquor in the gauge-glass remains constant, the volatile fatty acids are expelled with the water-vapor, but not completely. As the concentration of the liquor increases, the volatile fatty acids remaining combine

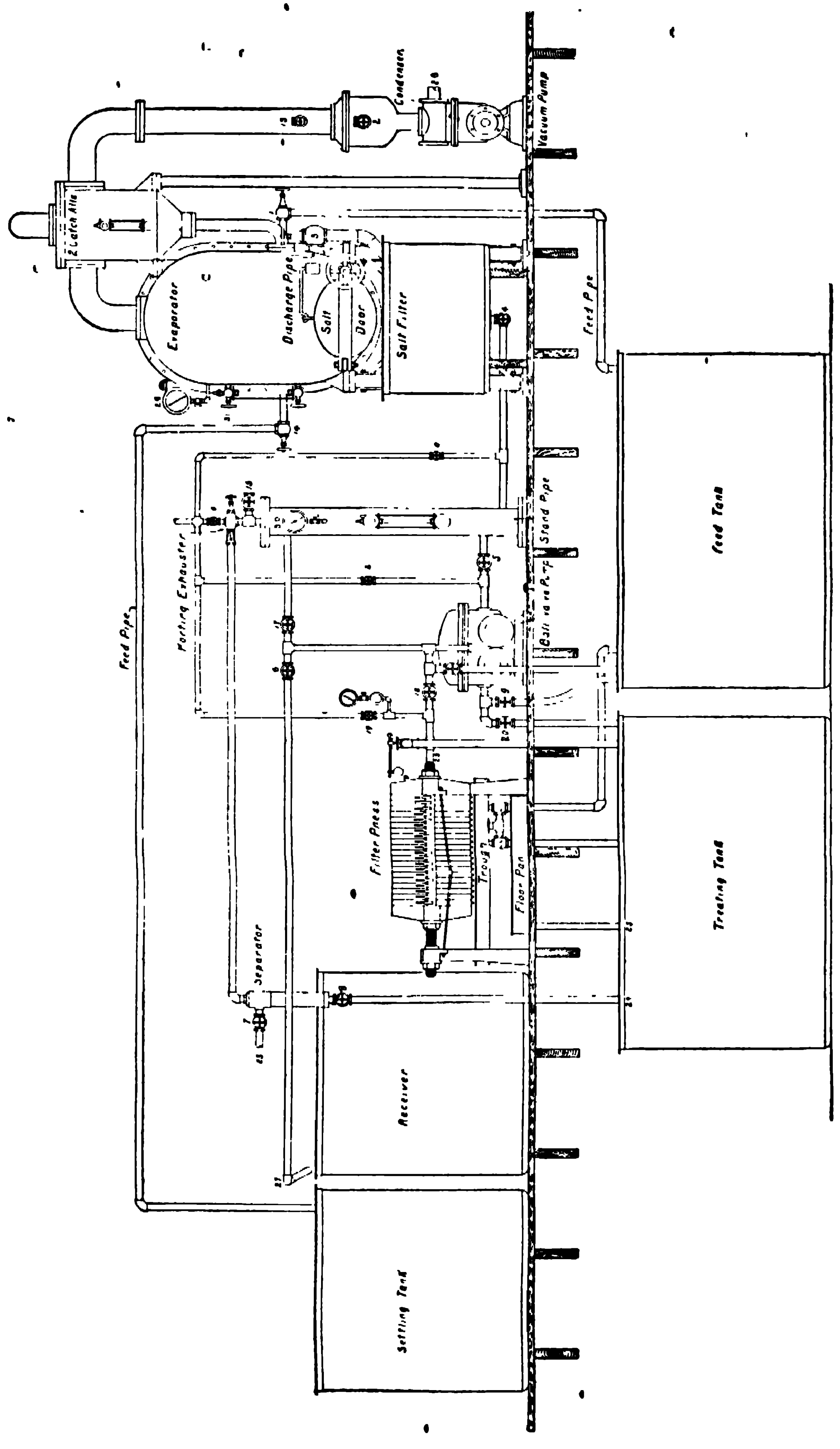


FIG. 216.—Crude Glycerin Plant—Garrigues System.

with glycerin, and are retained by it in the form of glycerides. When a satisfactory degree of concentration has been reached the contents of the evaporator are discharged, as before described, but at this stage the concentrated liquor is pumped to the receiver. This liquor is crude glycerin containing about 85 per cent of glycerin. It is tested for total acidity by titrating hot with standard caustic soda, titrating back the excess of alkali with standard sulphuric acid—just the converse of the original test of the total alkalinity of the waste lye. The amount of a saturated solution of soda-ash required for neutralization is calculated and the same is added to the charge of crude glycerin in the receiver, the amount being sufficient to render the mass slightly alkaline. The crude glycerin is now ready for distillation.

In the preceding description reference has been made to the use of but one evaporator, Fig. 215, and its appurtenant apparatus

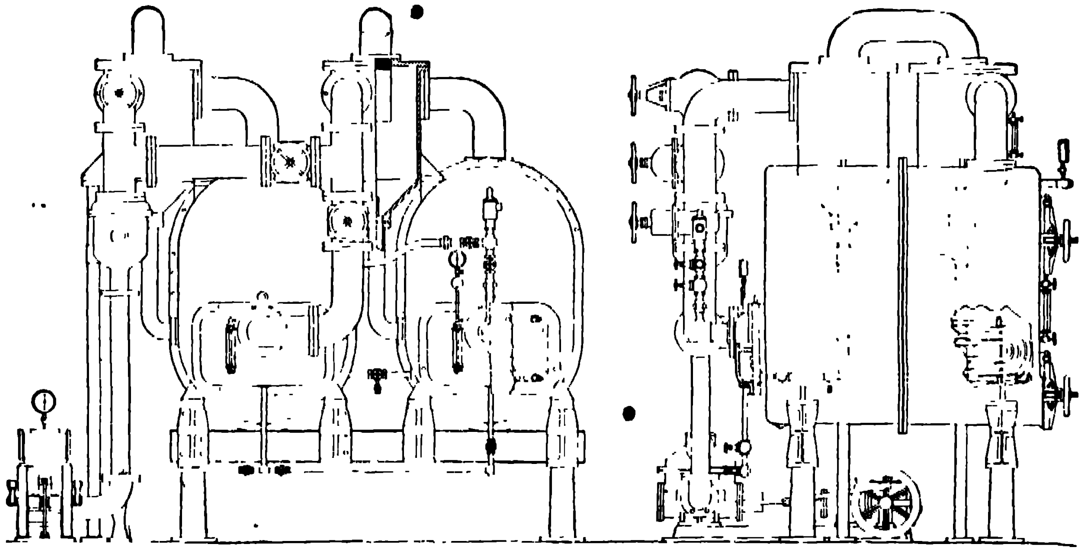


FIG. 217.—Garrigues Double-effect Evaporator, Convertible into Two Single-effects.

Fig. 216, in the production of half-crude and crude glycerin. In the factory installation two sets of apparatus are used, Fig. 217, one devoted solely to the production of half-crude, the other to the production of crude glycerin; thus permitting continuous and uninterrupted production of the supply of material for distillation.

Garrigues Double-effect Evaporator.—Inasmuch as the construction of the single-effect evaporator, the operation of which has been already described, and that of the double effect, which will now receive our attention, is practically the same, a description of the latter at this place will apply to both types.

The body of the evaporator is of cast iron, Figs. 218 and 219,

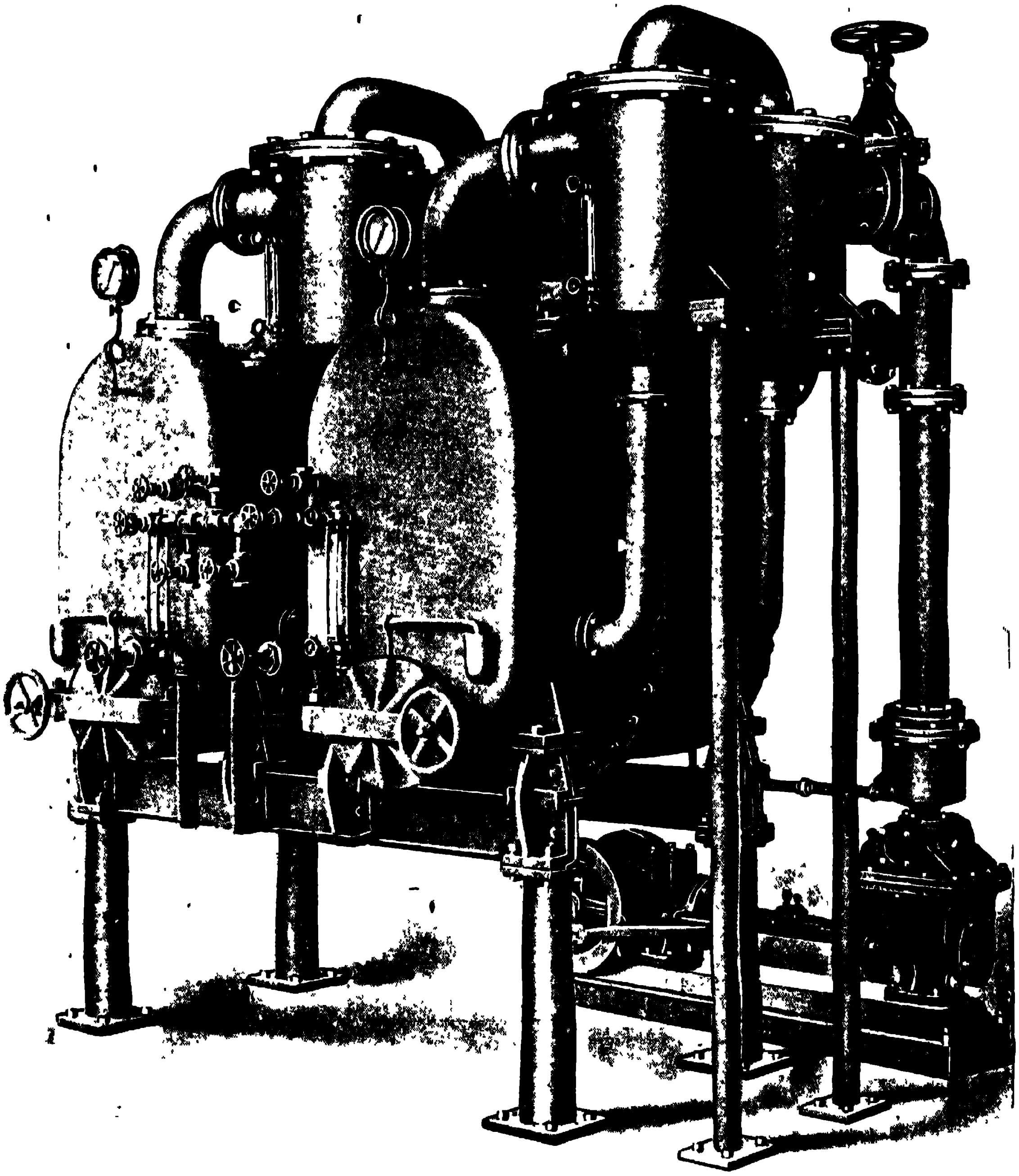


FIG. 218. Front View of Double-effect, Convertible, Vacuum Evaporator.

with brass U-shaped heating-tubes supported horizontally within the evaporator, as shown in Figs. 215, and 217, and free to expand in the direction of the bends.

The liquor to be evaporated surrounds the tubes, which are raised high enough from the bottom to make a chamber for the deposition of the separated salt. A door at the end serves for the removal of the salt at the end of the run.

The upper and lower legs of the U shaped heating-tubes connect

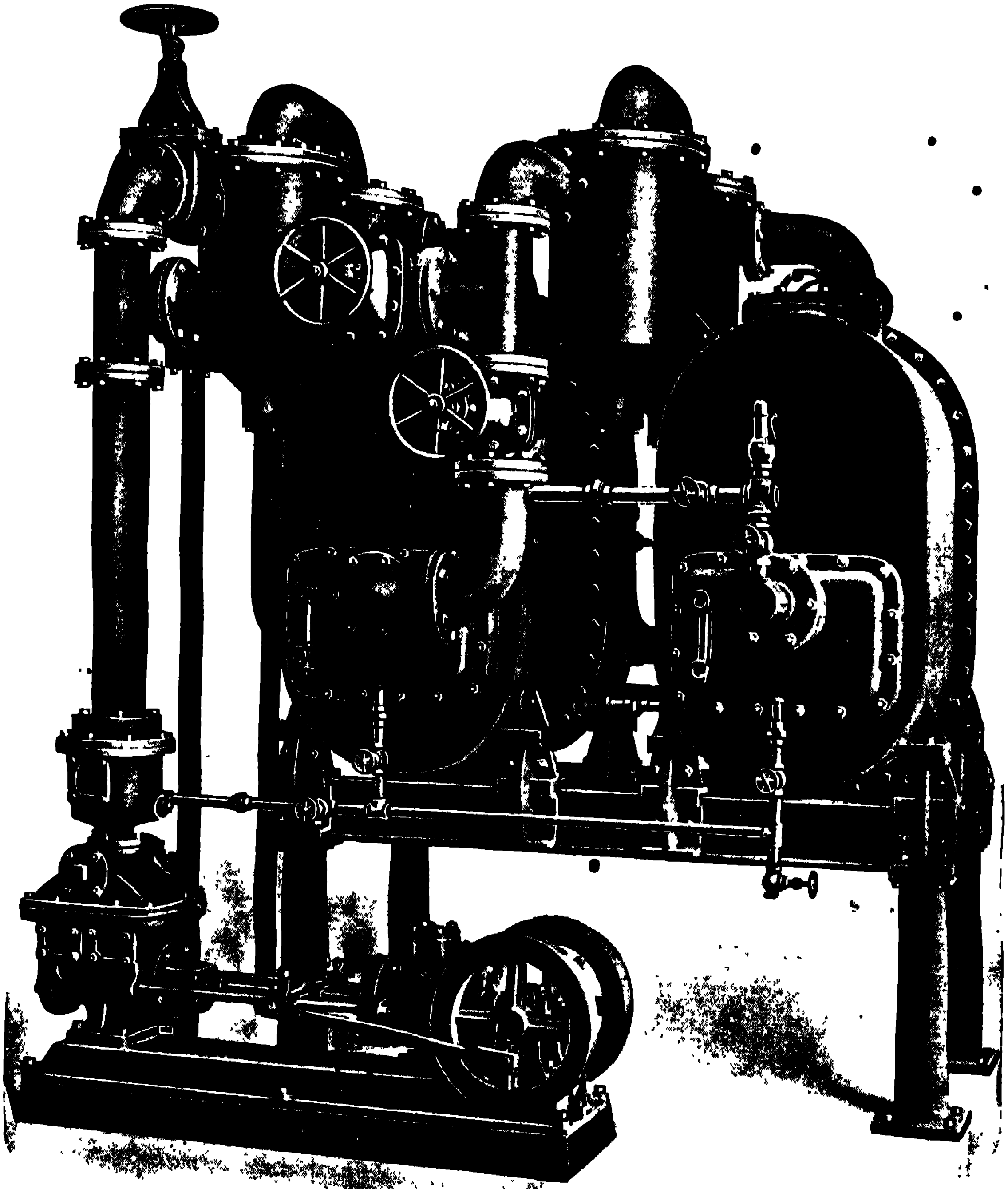


FIG. 219.—Rear View of Double-effect, Convertible, Vacuum Evaporator.

with separate chambers, as shown in section in Figs. 215 and 217 and in perspective in the rear view of the evaporator in Fig. 219, whereby the steam enters the upper chamber and traverses the upper legs, while the water of condensation formed in the lower legs is discharged into the lower chamber, where it is removed to a steam-trap, or to a pump if it is desired to return the hot condensed water to the boilers. Each effect is provided with two catch-alls, through each of which in succession the vapor must pass and which effectually returns any entrained liquor to the effect from which it came.

The double-effect evaporator may be used as two single effects working respectively on half-crude and crude glycerin, or as a double effect for the production of half-crude in the first instance.

In the reduction of purified waste lye to half-crude glycerin the apparatus is worked as a double effect in the following manner, referring to Figs. 217, 218, and 219.

Open the large valve on the vapor-pipe from the first effect to the steam chest of the second effect; also from the second effect to the condenser or vacuum-pump. Close the large valve on the cross vapor-pipe and open the drain-pipe from the steam-chest of the second effect to the vacuum-pump. Start the vacuum-pump with a little water to the condenser, which will exhaust the air from both effects. The three small valves between the two effects at the salt-door end of each evaporator are respectively the feed for waste lye, the feed for half-crude, and the vacuum-breaker. Open the feed for waste lye and charge both effects until filled to the middle of the sight-glass, which level is above the top of the heating-tubes. Turn on steam, provision being made for both live and exhaust steam. The lower large opening in the steam; chest (see right-hand effect, Fig. 219) goes only to the first effect, the smaller pipe above leading down into the steam-chest is for live steam. The latter also has a branch to the second effect, entering the large vapor pipe below the valve and thus leading to the steam-chest of the second effect.

When the liquor in the first effect begins to boil, the vacuum begins to fall until by the time the contents of the second effect begin to boil, the vacuum in the first effect is only about 18 inches as compared with about 26 inches in the second effect. From this stage until the end of the run the vacuum in the first effect will gradually fall until, with steam at one pound pressure in the tubes, about 12 inches are registered at the end. The vacuum in the second effect will remain fairly constant or may increase as evaporation proceeds and the liquor boils more slowly.

With the contents of both effects boiling, the feed valve is again opened, but now the feed is cut off from the second effect by an angle-valve on the feed-pipe entering that effect, a similar angle-valve on the feed-pipe to the first effect being checked in order to maintain a constant level therein. The second effect is fed from the first effect through the small pipe bearing a valve and connecting the two effects at their adjacent sides, this valve also being checked in order to maintain a constant level in the second effect.

Now check the drain-pipe from the steam-chest of the second effect leading to the vacuum-pump in order that only water of condensation may be removed from the steam chest. This water must be discharged to the vacuum-pump because there is the same degree of vacuum in this chest as in the first effect and the water would not discharge against atmospheric pressure. If it is desired to save this water it may be removed with a separate pump. The drain from the steam-chest of the first effect is discharged to a trap or feed pump, there being always a steam pressure at that point.

With the apparatus manipulated as described, evaporation should proceed regularly and at about the same rate in both effects, the second effect being heated by vapor from the first and vapor from the second effect being discharged to the condenser. The vacuum-pump maintains a vacuum in the second effect, while the partial vacuum in the first effect is due to the condensation of the vapor from the first effect in the heating-tubes of the second effect. Therefore the vacuum in the first effect falls as evaporation progresses, inasmuch as fresh lye is fed solely to the first effect and all the concentration takes place in the second effect. As the density of the liquor in the second effect increases, its boiling-point increases and its condensing power on vapor from the first effect is correspondingly reduced.

The economy of double-effect evaporation, wherever it is practicable, arises from the fact that only about 2 per cent. more heat is required to evaporate water in a vacuum of about 16 inches as compared with a vacuum of 26 inches; and the temperature of the vapor arising from the liquor boiling in a vacuum of 16 inches is ample in turn to boil the same liquor in a vacuum of 26 inches. In round numbers, a pound of steam admitted to the steam-chest of the first effect will evaporate a pound of water in that effect and the vapor from this water in turn will evaporate another pound of water in the second effect; so that evaporation by double effect consumes approximately about one half as much steam for the same work as evaporation by single effect. Loss of heat by radiation and in the water of condensation from the steam chest of the second effect reduces this proportion to a slight degree only.

On the completion of the run the liquor in the second effect will have attained a glycerin content of about 40 per cent. Steam is then turned off and the vacuum is broken. Separation of salt is confined to the second effect, the density of the liquor in the first effect not having reached the salting-point. At this stage in the double-effect evaporation of waste lye, the second effect is manipulated as the single effect previously described.

In the reduction of half-crude to crude glycerin it is inadvisable to use the double-effect system inasmuch as the separation of salt in the first effect seriously interfers with efficient evaporation. When an ample supply of half-crude has been accumulated, each evaporater is operated as a single effect with this material. To effect this change, the valve on the cross vapor-pipe is opened and that connecting the steam chests of the two effects closed. Water of condensation from the steam chests of both effects is discharged to the trap or feed-pump, the drain-valve to the vacuum-pump being closed. There are now available two single-effect evaporators working with one condenser and vacuum-pump and each heated with live steam. These are operated precisely as the single effect already described for the production of crude glycerin from half-crude.

Garrigues Process of Glycerin Distillation.—This process of distillation by steam is claimed to be well adapted for such liquids as show a tendency to decompose partially when distilled directly or alone at normal pressure, as glycerin. Such liquids are distilled in a partial vacuum, whereby the boiling-point of the liquids is lowered in accordance with the degree of exhaustion. They may be distilled with steam or aqueous vapor, which carries over the distilling liquid mechanically at a lower temperature than the normal boiling-point, or both methods may be combined for the purpose of preventing a partial decomposition of the liquids. In many instances it is desirable to use superheated steam. This is done for the purpose of distilling higher-boiling liquids without partially decomposing them.

Usually it is not feasible to completely condense the water-vapor and the vapor from the distilling liquid separately. In such cases if the liquid is miscible with water the water must be evaporated from the distillate to recover the distilled product water-free.

By this process the heat of the vapor of the distilling liquid is utilized for superheating the aqueous vapor employed in the distillation. The use of heat derived from this source is claimed to be a great improvement, from the standpoint of economy, over that obtained from steam as used in the present methods.

Confining our attention first to the theoretical aspects of this process we will consider the apparatus used and its mode of operation, and will then consider the practical detailed procedure of distillation.

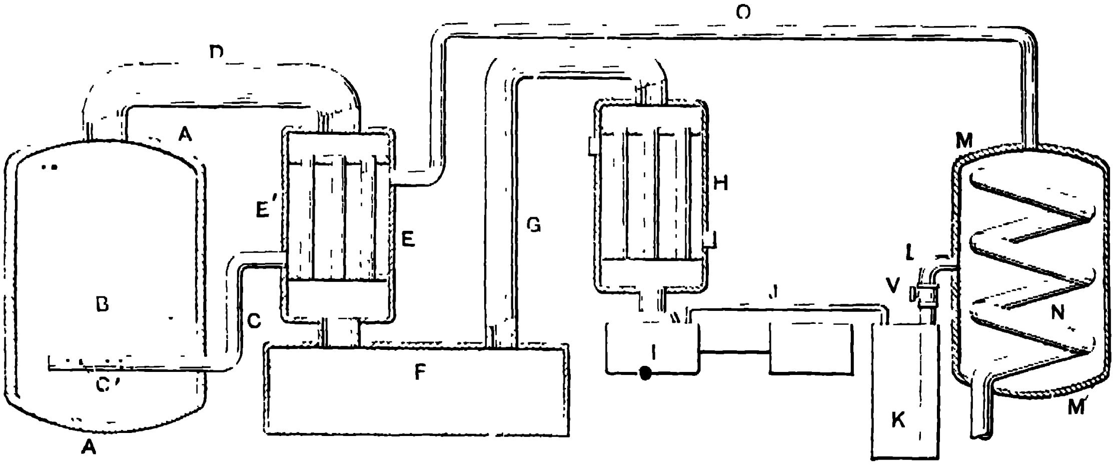


FIG. 220. Diagram of Garrigue's System for the Production and Distillation of Crude Glycerin.

Apparatus.—The essential parts of the apparatus by which this process is carried out are shown by the diagram in Fig. 220 wherein *A* represents the still containing the crude glycerin *B*, which, by virtue of the partial vacuum in the distilling apparatus, may be automatically fed into the still as required. The still may be heated either by means of a steam-jacket or a closed steam-coil immersed in the material to be distilled. These are not shown in the diagram. A pipe *C* for introducing superheated steam into the still extends within the still, and, as shown, is provided with perforations *C'*. At the top of the still there is a goose-neck *D*, which connects with the superheater and condenser *E* wherein the distilled liquor is condensed. The condenser connects below with the receiver *F*, from which a pipe *G* leads to a surface condenser *H* wherein the water-vapor passing through with some vapor of the compound distilled is condensed. This condenser is in connection with a vacuum-pump *I*. The discharge of the vacuum-pump connects, by means of a pipe *J*, with an open-tank *K*, through which a pipe *L*, with a regulating valve *V*, leads into the evaporator *M*. Within the evaporator there is a worm *N* which is heated by exhaust-steam from the vacuum-pump. At the top of the evaporator a pipe *O* leads over to the superheater *E*. On the lower part of the other side of this superheater the pipe *C*, which delivers the superheated steam into the still, is secured. From this diagram it will be seen that the water-vapor is in continuous circulation within the apparatus, thus making the process a continuous one.

Operation.—In the distillation of crude glycerin the liquid is fed automatically into the still, to which external heat is applied. The air is next exhausted from the entire apparatus by means of the vacuum-pump. The evaporator *M* contains sweet-water from a previous distillation which is heated by exhaust-steam from the vacuum-pump flowing through the coil *N*. Under the reduced pressure maintained in the apparatus by the operation of the vacuum-pump the sweet-water in the evaporator boils easily and vapor passes out through the pipe *O* into the superheater and condenser *E*. Here it passes around the hot condenser-pipes *E'* and is superheated by the hot glycerin and steam-vapors passing through them. From the superheater and condenser *E* the superheated steam passes through the pipe *C* into the still and is injected into the crude glycerin through the perforations *C'*. The steam thus injected causes the hot glycerin to distil over through the goose-neck. The glycerin-vapor mixed with steam-vapor passes now down through the condenser-tubes *E'*, located in the superheater. The water-vapors arising from the sweet-water in the evaporator pass simultaneously around these tubes and are superheated there, whereby the temperature of the hot condensing-vapors is reduced and the glycerin condensed, which then drops down into the glycerin-receiver *F*, where it is collected. As the sweet-water vapor is saturated steam and generated in a vacuum, its temperature is considerably below 212° Fahr. The vapors coming from the still, however, have a temperature of about 325° Fahr., the heat of the still being so regulated as to maintain the temperature of the vapors at about 325° Fahr. Accordingly there is a great difference of temperature between the condensing hot glycerin and steam-vapors coming from the still and the sweet-water vapors coming from the evaporator. Naturally the sweet-water vapors become superheated by passing around the condenser-pipes *E′* within the superheater and condenser *E*. The vapor-passage from the evaporator through the superheater *E* and perforations in tube or coil *C* must be of sufficient size to allow of maintaining practically the same vacuum in the evaporator and the still.

The heat of the vapors arising from the still is absorbed in the superheater by the water-vapors arising from the evaporator. This causes a condensation of the greater part of the glycerin within the condenser-tubes *E′*, from which it drops into the glycerin-receiver *F*, which latter may be a plain tank. The glycerin here obtained is a finished product. The water-vapor which passes along with the glycerin vapor, however, does not condense in the tubes *E′* of the superheater. It passes along through the tube *G* into the surface-condenser *H*, which is externally cooled by water in the usual manner. Within the tubes of the surface-condenser all the steam is condensed together with some glycerin-vapors carried along. This liquid is technically called sweet-water. From the surface-condenser the sweet-water drops down to the vacuum-pump and is removed by the latter to the open-top tank through the pipe *J*. From here the sweet-water passes into the evaporator, owing to the reduced pressure in the system by means of the evacuation. From the evaporator the water-vapors arise again and circulate through the system again in the described manner, whereby a continuous circulation is created and a continuous process obtained.

The small quantity of glycerin passing over with the water-vapor gradually accumulates in the sweet-water evaporator *M*. When the accumulation is sufficient to justify it the valve *V* is closed, stopping the feed of sweet-water to the evaporator until the water therein has been evaporated from its contents. The glycerin remaining in the evaporator is then removed and constitutes a finished product or may be further purified by redistillation.

By utilizing the water-vapors from the evaporator and superheating them by means of the hot vapors of the distilling glycerin and injecting the superheated vapor into the crude liquid, as described, a great saving is effected and at the same time a continuous process is obtained.

Distillation of Crude Glycerin, Garrigues System.—The theory of the Garrigues process for the distillation of crude glycerin having been described, we may now consider the practical procedure of distillation, referring for illustration to Fig. 221, in which is shown the construction and appurtenant apparatus of the still. Referring to the distilling-plant, Fig. 221, air is first exhausted from the entire system by means of the vacuum-pump. Crude glycerin is then introduced into the still through the feed-valve on the side, a gauge-glass (not shown) indicating the level of the contents, which is maintained steadily at a little above the perforated pipe in the still. The still having been charged, steam at upwards of 125 pounds pressure is admitted into the heating-coils of the still. In the meantime the sweet-water evaporator has been charged with sweet-water from the previous distillation, the same being drawn from the storage-tank. Low-pressure steam is supplied to the tubes of the evaporator for which purpose exhaust from the pump is available. The sweet-water evaporator is similar in construction to the crude evaporator. When the sweet-water is brought to boiling, the vapor therefrom passes through the pipe to the superheater and thence to the jet-pipe of the still and escapes through the charge of crude glycerin contained therein. The glycerin distills over, the catch-all, as shown, serving to return any entrained liquor to the still. The temperature of the vapor is maintained at about 320° Fahr., the corresponding temperature of the charge likewise being maintained by that portion of the heating-

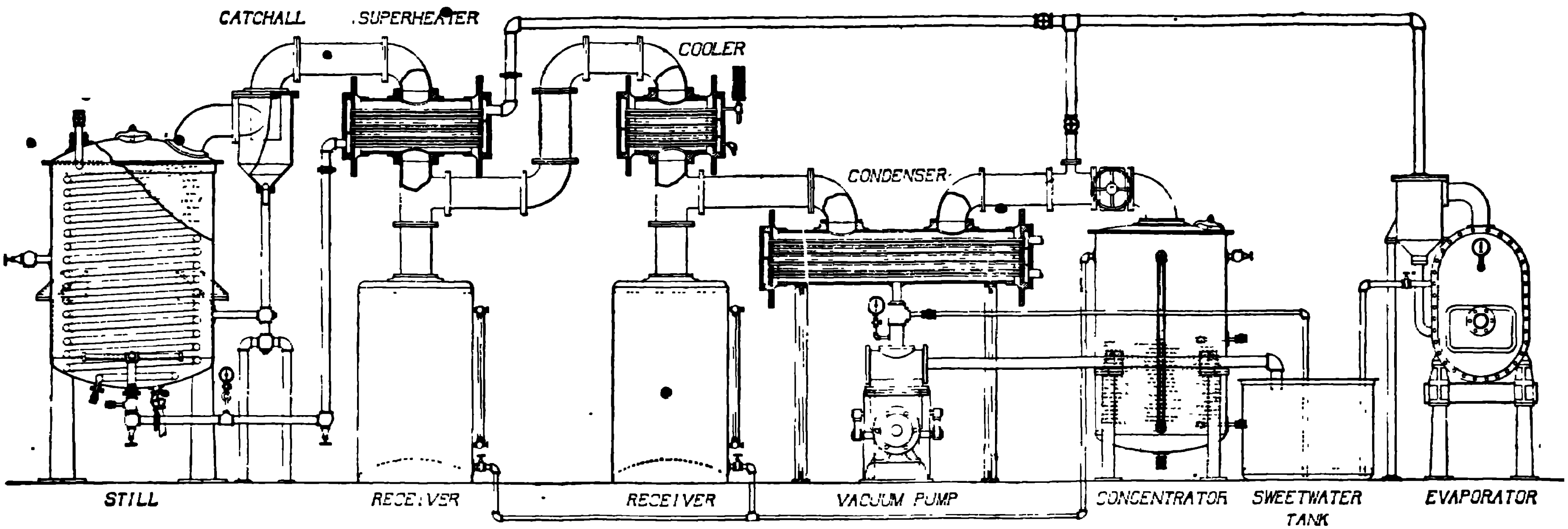


FIG. 221.—Complete Plant, Garrigues System, for the Production and Distillation of Crude Glycerin.

coils immersed in it. The hot vapor from the crude glycerin superheats the steam evolved from the sweet-water evaporator to the degree that it imparts to it about 160° Fahr. of "superheat," so that it is delivered into the still at a temperature of about 300° Fahr. and at a vacuum of 24 inches.

The abstraction of heat from the vapor of the distilling glycerin condenses a large part of the glycerin on the tube surfaces of the superheater, which glycerin drops into the first receiver. It is almost water-white, with a density of about 1.263. The vapor now passes to the glycerin-cooler, where it comes in contact with the external surface of tubes through which warm water is circulated, the temperature being maintained above 150 Fahr., so that practically no water is condensed. The density of the condensed liquor is about 1.250. The temperature of discharge of the water in the tubes, as indicated by a thermometer in the outflow, is maintained at about 190° by using more or less water feed as the conditions require. It is not desired that all the glycerin condense here, otherwise the volatile fatty acids which have escaped elimination in the crude glycerin-evaporator would also condense and impart to the distillate a too high acidity. It is advisable for this reason to operate so that about 3 per cent of the total glycerin will pass into the sweet-water condenser, where it, with all the steam that has been injected into the still, will be condensed by cold water circulated inside the tubes.

The best distilling conditions are represented by the proportion of two pounds of steam injected into the still per pound of glycerin distilled. Hence if the sweet-water contains 3 per cent of the total glycerin it should have a glycerin content of approximately 1.5 per cent. It is pumped from the condenser as fast as it accumulates by means of the vacuum-pump, which discharges it into an open tank. As there is usually not sufficient sweet-water to keep a pump properly sealed, it is advisable to have a small return-pipe, as shown in Fig. 221, from the open tank to the pump-suction, so as to permit some of the sweet-water to maintain a continuous circuit through the pump. From the open tank the sweet-water is fed into the evaporator, as already described, where there is maintained a vacuum of about 22 inches. The glycerin content of the sweet-water increases as evaporation proceeds; as the concentrated sweet-water possesses considerable acidity redistillation is necessary for the preparation of dynamite or the chemically pure grades of refined glycerin.

Inasmuch as the crude glycerin obtained by the concentration of sweet-water will contain about 9 per cent of water as compared with about 1 per cent in the "dynamite" glycerin, and as no water is lost in the circuit of operations, there will be a constant increase of the volume of sweet-water in circulation. For each pound of glycerin contained in the sweet-water, about 3 pounds of glycerin are obtained from the crude glycerin distilled, so that the density of the liquor in the sweet-water evaporator will not exceed that corresponding to a glycerin content of about 25 per cent. This liquor may be finally evaporated to an 80 per cent crude to be redistilled by shutting down the still, closing the valve on the pipe leading from the sweet-water evaporator to the still and opening the valve on the pipe connecting the said evaporator with the sweet-water condenser. The aqueous vapor condensed during this evaporation is discharged to waste by the vacuum pump. A new run of the still may then be started by feeding ordinary water to the sweet-water evaporator until sweet-water begins to come over. However, it is better practice to empty occasionally the contents of the sweet-water evaporator into the settling-tank of the crude glycerin plant and to feed the same into the still as required for distillation.

In the operation of the still the best practice is to maintain the feed of crude glycerin at a constant level in the still, viz., just above the perforated jet-pipe, during the entire duration of the distilling period, which may be as long as 24 hours. The charge then remaining in the still is withdrawn and transferred to the waste-lye treatment-tank, where the coloring-matter is again removed by chemical treatment and filtration as before described.

Joslin Apparatus for Distillation of Crude Glycerin.—Fig. 222 is a compound view of the glycerin distilling apparatus covered by the patent of Joslin and Schmidt; the heating-coils being shown in plan and other parts partly in side elevation and partly in central vertical section. *A* represents the outer shell of the still, which is supported upon feet *a*. The part *A* has outwardly-projecting flanges a^1 upon each end to form seats for the heads a^2 and a^3, which are secured to the shell *A* by bolts or screws passing through the heads and tapped into the outwardly-projecting flange. The shell *A* has cast integral with it an interior cylinder A^1, which is connected to the outer cylinder by partitions a^4 and also inwardly-projecting baffle-plates a^5. The heads a^2 and a^3 have perforated bosses a^6 and a^7, cast integral with the heads. These bosses form bearings for the hollow journals of the inner revolving cylinder *B*, one of the protruding journals being provided with a gear-wheel *b*, which meshes with the pinion b^1, secured upon a driving-shaft b^2, which shaft is also provided with tight and loose pulleys b^3 and b^4, which are driven by a belt.

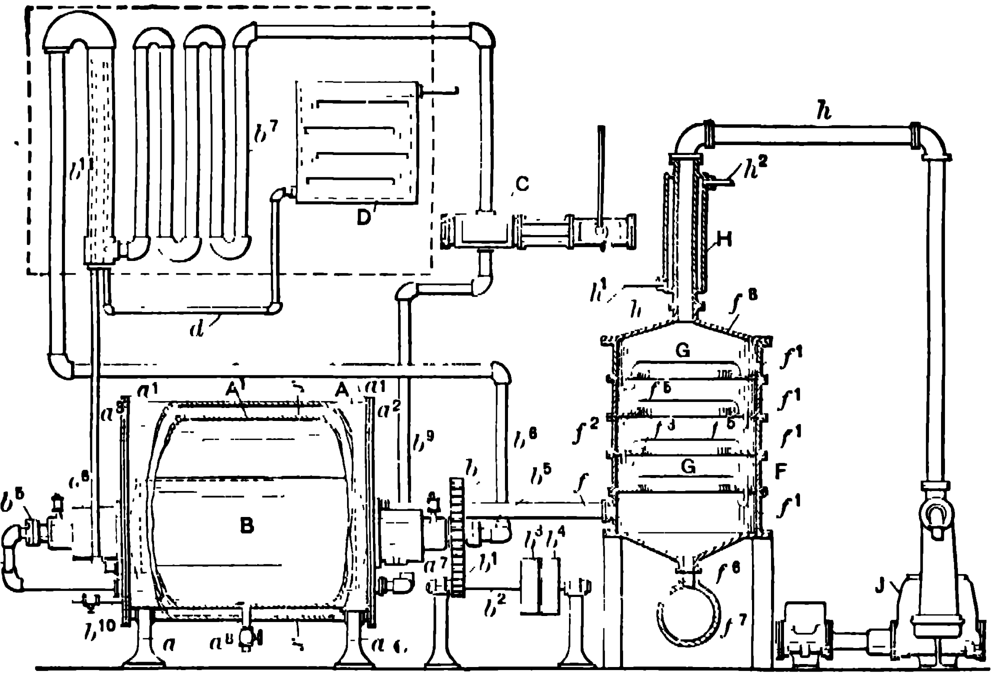


FIG. 222.—Glycerin-still (Joslin System).

The hollow journals of the cylinder *B* have swivel-couplings b^5 upon their ends, to the inner one of which is connected the pipe b^6, which conducts the heating agent (preferably paraffin oil) from the heating-coil b^7 to the interior of the cylinder, and the other receives the pipe b^8, which conducts the heating agent from the cylinder to the jacket or chamber between the outer shell *A* and the inner shell A^1 and the partition a^4. The heating agent is carried from the jacket through the pipe b^9 back to the coil b^7 to be reheated, by pump *C*.

The cylinder *B* revolves in the glycerin, which is supplied to the bottom of the cylinder A^1 through a pipe b^{10}, the amount being regulated by a cock interposed in the pipe. The purpose of the lower jacketed space between the partitions b^4 is to prevent the heating agent from coming in contact with that part of the cylinder A^1 which contains the glycerin and forming a scale or decomposing and burning the liquid to be distilled. The liquid to be distilled is carried up in a thin film around the revolving cylinder and is met by a current of steam, which is superheated by passing through a cast-iron coil *D*, thence through the pipe *d*, which has a return-bend within the terminal b^{11} of the coil b^7. The pipe *d* enters the cylinder by means of suitable couplings through the head a^3, and the part of it within the cylinder A^1 is perforated along its entire length to discharge the steam against the cylinder and downwardly in the direction of the liquid to be distilled. By placing the perforated pipe in the position shown the jets of steam are discharged above the liquid in the still, by which the foaming so objectionable is entirely prevented.

The purpose of the baffle-plates a^5 is to interrupt the current of steam which is admitted into the cylinder A^1 and deflect it against the liquid carried up on the revolving cylinder, and the purpose of placing the steam-pipe inside of the terminal coil, through which the heating medium for the still passes, is to bring the steam to the same temperature as the coil or other heating medium before it enters the cylinder *A*.

In order to present a clean bright surface to the revolving cylinder which carries up a film of the liquid to be evaporated, there is provided a thin blade or scraper *E*, which is mounted upon a rod secured in the cylinder heads and bears with spring-pressure against the periphery of the revolving cylinder *B* throughout its full length, thus preventing the formation of any scale or sediment upon the periphery of the revolving cylinder. Any sediment from the glycerin settling in the bottom of the cylinder A^1 may be drawn off or discharged through the pipe a^8.

In the still the glycerin carried up by the revolving cylinder is distilled and mingles with the superheated steam and both distilled over together through the pipe *f* to the bottom section of the separator *F*. The separator is composed of a series of flanged sections

f^1 each section having outwardly-projecting flanges, by means of which the sections are bolted together to form the separator F. Each section has an inwardly-projecting shelf f^2, which, with its upwardly-extending flange f^3, forms an annular trough around the inner wall of the section. Each section is provided with a cap G, which extends over the inner wall of flange f^3 of the gutter and is supported above the shelf f^2 by feet or angle plates f^4, which are riveted upon the shelf and to the downwardly-curved flange of the cap G. Through the shelf of each section, but on alternate sides, is a drip-pipe f^5, the upper end of which extends to a plane slightly below the upper edge of the flange or inner wall of the gutter f^3 and above the lower edge of the cap G to form a seal and prevent the escape of the vapor after passing through the glycerin in the shelves. The distillate entering the lower section is deflected by the cap G into the gutter. The glycerin being the heavier substance fills the gutter up to the top of the pipe f^5. The unseparated vapors passing to the section above are again conveyed to the gutter until all the gutters are filled up to the top of the pipe f^5 with the separated glycerin, after which the vapors bubble through the layer of liquid in the troughs and the separated glycerin overflowing from one trough to the other drips into the cone shaped bottom of the separator and is then from there conveyed through a pipe f^6 to the receiving-drum f^7. To the top of f^8 of the separator is secured the condenser H, which consists of the pipe h, through which the escaping steam from the separator is drawn, as shown, by the vacuum-pump J. The condenser is the cylinder H, surrounding this pipe h, and through the jacket between the pipe and outer shell of the condenser a stream of cold water is run, entering at the pipe h^1 and passing out through the pipe h^2. The object of the condenser is to condense a portion of the escaping steam and return it to the top plate G of the separator to keep it cool. The upturned bends of the pipes f^5 serve as traps to prevent the vapor from passing up through them.

The heating-coils are arranged in a furnace-wall (indicated in dotted line) with a grate below to receive the fuel and an arrangement (not shown) by which the heat can be controlled, so that the heating medium when it enters the revolving drum can be kept at any desired temperature.

Operation.—The operation of the apparatus is as follows: The heating medium for the revolving drum (preferably paraffin oil) is heated by the fire in the furnace, and the circulation is supported by means of the pump *C*, first through the heating-coils, then through the revolving drum, thence through the jacket around the drum, and back again to the heating-coils. The fire in the furnace is so controlled that the temperature of the heating medium as it enters the revolving drum is that found most suitable for the liquid to be distilled. The liquid to be distilled is now run into the cylinder A^1 forming a shallow layer at the bottom thereof. The drum revolving carries a thin film of this liquid up, at the same time heating it to the distilling-point. The steam, which is preferably superheated to the temperature of the heating medium by passing through the coil *D* and the terminal of the coil b^7, passes in to the interior of the still over the surface of the drum, the contact of which is facilitated by the baffle-plates a^5, and escapes, thoroughly saturated with the vapors of the distillate, through the pipe *j* to the separator *F*. This vapor, entering the lower section of the separator, passes through the various sections from the bottom to the top through the successive compartments. During this passage the vapor is gradually deprived of the valuable parts of the distillate until it leaves at the top, steam only escaping through the condenser *H*. A small portion of the steam only is condensed in the pipe *h* and flows back to the top of the upper plate, thus preventing the escape of any glycerin.

By means of this arrangement a distillate of any desired gravity can be drawn off from the bottom of the separator.

Yield of Glycerin.—The practical yield of refined glycerin from stock saponified depends upon varying factors, which have already been mentioned, and will range from 60 to 85 per cent of the theoretical yield. The yield of concentrated products from clarified waste lye depends upon the percentage of glycerin contained therein. Crude glycerin of good quality should yield on distillation 80 to 90 per cent of half-refined glycerin; half-refined should yield on second distillation an amount of refined depending on the density of the half-refined glycerin. The yield of glycerin is estimated on the basis of stock killed; as stock yields in round numbers twice its weight of waste lye, figures thus derived from a stock-basis are practically halved for waste lye.

Glycerin: Origin, Composition, and Properties.—Although glycerin may be found by chemical analysis in small amount mixed with glycerides that have undergone more or less spontaneous decomposition, and although it is produced in small quantities during the alcoholic fermentation of sugar, it does not exist in nature as a distinct and separate body. It is a product of artifice and is formed from the elements of water and the basic triatomic portion of salts of fatty acids when the salts are decomposed either by caustic soda in soap-manufacture, in accordance with the following equation:

$$\begin{array}{rlclrl}
\multicolumn{2}{c}{\text{One Molecule of Glyceryl Stearate or Stearin.}} & & \text{Three Molecules of Caustic Soda.} & \multicolumn{1}{c}{\text{One Molecule of Glyceryl Hydrate or Glycerol.}} & \text{Three Molecules of Sodium Stearate or Soap.} \\
 & O\cdot C_{18}H_{35}O & & NaOH & OH & Na\cdot C_{18}H_{35}O_2, \\
C_3H_5 & O\cdot C_{18}H_{35}O & + & NaOH & = C_3H_5 \, OH & + Na\cdot C_{18}H_{35}O_2, \\
 & O\cdot C_{18}H_{35}O & & NaOH & OH & Na\cdot C_{18}H_{35}O_2,
\end{array}$$

or by water as employed in the decomposition processes applied to stock in candle-manufacture and which may be represented by the following equation:

$$\begin{array}{rlclrl}
\multicolumn{2}{c}{\text{One Molecule of Glyceryl Stearate or Stearin.}} & & \text{Three Molecules of Water.} & \multicolumn{1}{c}{\text{One Molecule of Glyceryl Hydrate or Glycerol.}} & \text{Three Molecules of Stearic Acid.} \\
 & O\cdot C_{18}H_{35}O & & \left\{\begin{matrix} H\cdot OH \\ H\cdot OH \\ H\cdot OH \end{matrix}\right\} & OH & H\cdot C_{18}H_{35}O_2 \\
C_3H_5 & O\cdot C_{18}H_{35}O & +3 & & = C_3H_5 \, OH & + H\cdot C_{18}H_{35}O_2. \\
 & O\cdot C_{18}H_{35}O & & & OH & H\cdot C_{18}H_{35}O_2
\end{array}$$

In soap-manufacture, according to the molecular weights of the reacting bodies, 890 parts of stearic acid combine with 120 parts of sodium hydrate to form 92 parts of glycerol and 918 parts of soap (in the explanatory equations the soap represented is that of stearin, typical of tallow), this being equivalent to a yield of 10.34 parts of glycerol and 103.14 parts of soap (sodium stearate) from 100 parts of stearin (tallow), indicating that the yield of products above the equivalent weight of soap is due to the absorption of the elements of sodium hydrate. The hydrogen of sodium hydrate has united with the base glyceryl to form glycerol, while the remaining elements have combined with the liberated fatty acid to form sodium stearate (soap).

In candle-manufacture, according to the molecular weights of the reacting bodies, 890 parts of stearin combine with 54 parts of water to form 92 parts of glycerol and 852 parts of stearic acid, this being equivalent to a yield of 10.34 parts of glycerol and 95.73 parts of stearic acid from 100 parts of stearin, indicating that the yield of products above the equivalent weight of stearin is due to absorption of the elements of water. The hydroxyl, OH, of water has gone into stearic acid, while the remaining hydrogen, H, has combined with the base glyceryl to form glycerol.

Glycerin is a triatomic alcohol, hence in strict allegiance to chemical phraseology it should be called glycerol, the term glycerin being applied to its commercial forms.

The composition of crude glycerin has been already described Half-refined glycerin, which is the product of the distillation of crude glycerin, is of varying straw-color and density according to the quality of the crude and the care used in refining. Refined glycerin, which is the product of the distillation of half-refined glycerin, is of pale-straw color, of varying composition and density and when below 1.26 in specific gravity at 15° C. is usually concentrated. In the following table are shown actual factory analyses of refined glycerin of dynamite grade which will serve to indicate the variation in composition of superior products of this grade.

TABLE XXXVII.—ANALYSES OF DYNAMITE-REFINED GLYCERIN FROM WASTE SOAP LYE.

Sample.	Per Cent NaCl.	Per Cent Ash.	Per Cent Carbonaceous Residue.	Per Cent Acidity Estimated as Na_2O.		Specific Gravity at 15° C.
				Free.	Comb.	
Maximum Limit.	0.002	0.005	0.05			
1	0.001	0.004	0.014	....	0.0058	1.2623
2	0.001	0.0012	0.026	....	0.020	1.2623
3	0.001	0.0036	0.008	0.0029	0.013	1.2629
4	0.001	0.0008	0.006	0.0058	0.029	1.2628
5	0.001	0.0012	0.008	....	0.040	1.2625
6	0.001	0.0008	0.004	....	0.021	1.2628
7	0.001	0.0020	0.004	....	0.0179	1.2628

The salt is the last residue of that originally used to grain the soap. Ash comprises salt, sodium sulphate, and sodium carbonate formed by the ignition of sodium salts of fatty acids of undetermined composition. Carbonaceous residue comprises non-glycerin matter, the organic portion of which constitutes the free and combined acidity which is expressed in terms of sodium oxide, Na_2O. Dynamite glycerin discolors more or less on aging, according to the percentage of organic impurities not removed in the refining process.

Colorless glycerin of the pharmacopœia obtained by distillation and clarification from the dynamite grade is when of 1.265 specific gravity at 15° C., a pure product. The British pharmacopæia (1898) requires of pure glycerin the following tests: It must be a clear, colorless, syrupy liquid of a sweet taste, inodorous, miscible with water and alcohol (90 per cent), neutral to litmus, insoluble in ether, chloroform, and fixed oils. Its specific gravity should be 1.260. It should yield no characteristic reaction with the tests for lead, copper, arsenium, iron, calcium, potassium, sodium, ammonium, chlorides or sulphates, and no red precipitate with excess of solution of potassic-cupric tartrate on boiling, even when previously acidified and boiled. It should undergo no darkening in color at ordinary temperatures when mixed with an equal volume of solution of ammonia and a few drops of solution of silver nitrate; and when shaken with an equal volume of sulphuric acid (the mixture being kept cool) no coloration, or only a very slight straw coloration should result. When gently heated with a mixture in equal volumes of alcohol (90 per cent) and diluted sulphuric acid a fruity odor should not be produced. If 2 cubic centimeters be diluted with 5 cubic centimeters of a mixture of 1 part of hydrochloric acid and 7 parts of water, 1 grm. of pure zinc being added, and the whole placed in a long test-tube, the mouth of which is covered by a piece of filter-paper moistened with a drop or two of test solution of mercuric chloride and dried, it should not afford a yellow stain on the paper, even after fifteen minutes. When it is evaporated in an open capsule it should not leave any ash.

Perfectly pure glycerin is a solid crystalline substance melting at 17° C. [22° C.] [36° C.], but the merest traces of impurities prevent it from crystallizing. It is familiar as a thick, transparent, colorless and odorless liquid, with a very sweet taste. It has a specific gravity of about 1.266 at 15° C. By cooling glycerin to −40° C. Berthelot could obtain it as an almost solid gummy mass. Wm. Crookes gives an account in the *Chemical News* of Jan. 18, 1867, of 5 tons of glycerin imported into London from Germany in casks of 8 hundredweight each, which, though when it left the continent it was in its ordinary state of a viscid liquid, was found on reaching London to have become solidified to a mass of very hard brilliant crystals. The same result was observed in Vienna in a mass of glycerin which had been in an iron tank for more than a year (*Chemical News*, Apr. 5, 1867). The crystalline mass noted by Crookes yielded pure glycerin when melted. The same result was observed by the author and others in Boston in the winter of 1898. A wooden barrel of dynamite-refined glycerin had been returned to the factory by a consumer who declared it unusable owing to solidification. The crystals when melted were of the same density as the original liquid. Sarg obtained glycerin in crystals by long exposure to the cold. The phenomenon, however, is exceptional. Crystallization takes place slowly and is greatly promoted even between 0°–5° C. by the addition of a solid crystal. Glycerin boils at 290° C., and this boiling-point is so constant that it has been recommended by Th. Gerlach for the purpose of fixing this point on high-temperature thermometers.

According to Allen, however, a small addition of water, about 5 per cent, lowers the boiling-point to 164° C.

Under the action of dehydrating agents, or when distilled under atmospheric pressure, as noticed in the offensive odor on extinguishing the old-fashioned candle made of tallow, it decomposes into acrolein in accordance with the following equation:

$$\underset{\text{Glycerin.}}{C_3H_8O_3} = \underset{\text{Acrolein.}}{C_3H_4O} + \underset{\text{Water.}}{2H_2O}.$$

When pure and anhydrous it ignites at 150° C. and burns gently with a pale-blue, non-luminous flame without odor or residue. At a temperature of 320° Fahr. glycerin dissolves two-thirds of its weight of boric acid forming glyceryl borate $C_3H_5BO_3$, which has been patented and finds application as a preservative agent under the name "boro-glyceride." It unites with alkalis and alkaline earths to form compounds soluble in water; of its many derivatives nitro-glycerin is the most important. In contact with yeast and other ferments under suitable conditions it is decomposed with formation of propionic and butyric acids.

Glycerin is a most powerful solvent. It mixes in all proportions with water or alcohol, but is insoluble in ether, carbon disulphide, benzene, chloroform, and petroleum spirit. It dissolves many substances, such as iodine, carbolic acid, mercuric iodide, and the alkaloids, more easily than water does; it also dissolves sodium hydrate or caustic soda, oxide of lead, the sulphates and chlorides of potassium, sodium, and copper, all deliquescent salts, and the vegetable acids. Its great affinity for moisture adapts it to numerous uses in the arts. The water-absorbing power is so great that on exposure to the atmosphere glycerin will take up as much as 50 per cent of its own weight of water. In contact with the skin, especially the mucous membrane, the sensation of heat is produced from the abstraction of moisture.

The powerful solvent properties of glycerin, combining in this respect the properties of alcohol and water, and thereby permitting the solution of many substances with greater ease in glycerin than in either water or alcohol alone, are shown in the following table:

100 parts of glycerin dissolve	98	parts of	crystal soda
	60	" "	borax
	50.5	" "	potassium arsenate
	50	" "	sodium arsenate
	50	" "	zinc chloride
	40	" "	alum
	40	" "	potassium iodide
	30	" "	copper sulphate
	25	" "	ferrous sulphate
	25	" "	potassium bromide
	20	" "	lead acetate
	20	" "	ammonium carbonate
	20	" "	arsenious acid
	20	" "	arsenic acid
	20	" "	ammonium chloride
	15	" "	oxalic acid
	10	" "	barium chloride
	10	" "	copper acetate
	8	" "	sodium carbonate
	7·5	" "	mercuric bi-chloride
	5	" "	calcium sulphide
	3.5	" "	potassium chlorate
	1.9	" "	iodine
	1	(about) part of	calcium sulphate
	0.1	part of	sulphur

Friedel and Silva effected its synthesis from propylene chloride, which in turn was made without the use of glycerin. Berthelot has combined it under suitable conditions with fatty acids and thus produced the glyceride.

Nitro-glycerin.—The use of glycerin in the manufacture of nitro-glycerin, which in turn forms the basis of dynamite and the high explosives used in engineering, absorbs the greater part of the production of this body. Its use in the manufacture of numerous other commodities is increasing and new uses are daily being found. However great the consumptive outlet in this direction may be, the use of glycerin in the manufacture of explosives will remain the chief and primary one and will determine its value in the markets of the world.

The interest of soap-manufacturers in the recovery of this body will extend to its first use as the raw material in the production of nitro-glycerin. The requirements of refined glycerin for nitration in the manufacture of nitro-glycerin are as follows: It must be of light-yellow color and of specific gravity of not less than 1.261 at 15° C.; CaO, MgO, Al_2O_3 and Fe_2O_3 must be absent; it must not become milky with $AgNO_3$ solution indicating presence of chlorides; only minute traces of arsenic are tolerated; and it must contain no organic impurities beyond traces of lower fatty acids. The most satisfactory test of a refined glycerin for this purpose is the yield obtained on subjecting the sample to nitration.

Nitro-glycerin is manufactured by mixing glycerin with a mixture of nitric acid and sulphuric acid. Each of the materials used is the most concentrated that can be made, and the demand for large quantities of nitric and sulphuric acids and glycerin of the highest grades which has been created by the high-explosives industry has had a marked effect on the development of the acid and glycerin industries. The acids are usually mixed in the proportion of 3 parts by weight of sulphuric acid to 2 parts by weight of nitric acid, and they should contain 61.9 per cent of H_2SO_4 and 34.5 per cent of HNO_3, with not more than 0.7 per cent of N_2O_3. These previously mixed acids are sent out from the acid works in iron drums holding about 1500 pounds, and this weight of mixed acids makes a convenient charge for one run in the nitro-glycerin converter, from 210 to 230 pounds of glycerin being there mixed with it. The reaction goes on between the glycerin and the nitric acid, the sulphuric acid present serving chiefly to take up and retain the water which is one of the products of the reaction. When the reaction is completed the materials are run into a tank, where they rest, until owing to their differences in specific gravity, the nitro-glycerin and spent acids form into separate layers; then the nitro-glycerin is run off into washing and purifying-tanks, and the acids are run off to be reworked. The dilute nitric acid thus obtained is sometimes used in the manufacture of ammonium nitrate for use in dynamite dopes. The diluted sulphuric acid is sometimes used in the manufacture of nitric acid, but is more often concentrated in iron pans, and, after being mixed with strong nitric acid, again used in making nitro-glycerin. This spent acid averages in composition 72 per cent of sulphuric acid, 10 per cent of nitric acid, and 18 per cent of water. Theoretically 100 parts by weight of glycerin should yield 246 parts of nitro-glycerin, but in practice the yields are from 200 to 220 parts.

Nitro-glycerin is used directly in torpedoes, which are cylinders holding 20 quarts each, for "shooting" oil wells. It also is used in medicine as a heart stimulant. The principal use of nitro-glycerin is in the manufacture of dynamite and blasting gelatin.

Uses of Glycerin.—Glycerin is used in the arts either alone in virtue of its distinctive properties, or as a solvent possessing wide range and powerful capacity, or as a substitution product. In medicine glycerin is used chiefly as a vehicle for applying externally many substances to the skin. If applied undiluted it absorbs water from the skin, but in dilution its high penetrative power and affinity for moisture adapt it well for external application. It is used as a lotion, or as a basis for ointments in the treatment of wounds, sores, and bites of venomous insects. Large internal doses are liable to cause loss of muscular energy, lethargy, and even death. It is a constituent of a number of officinal fluid extracts. It is used for many purposes in pharmacy not only to prevent drying but decomposition as well.

Its chief use in the arts is as a nitro-substitution product in the manufacture of heavy explosives. It is used in gas-meters to prevent freezing; in hydraulic jacks for the same purpose; to lubricate interior of moulds; to prevent the shrinkage of wooden vessels; in clay-modeling and in artists' colors to maintain softness; as a lubricant of delicate machinery; to preserve meats, fruits, candies, mustard and medicine; as an ingredient and preservative of tobacco; in soaps and cosmetics; in the extraction of perfume from flowers; to impart elasticity to paper; in the preparation of leather; in photography; in the construction of mercurial manometers; as a solvent for certain aniline colors; in the preservation of anatomical and microscopical preparations; in calico-printing; to prevent the rusting of instruments; in the production of artificial oil of mustard; in the preparation of certain cements, and of copying-ink and blotting-pads; in the manufacture of printers' rollers; in rubber substitutes, and in brewing. The above list, while by no means intended to be complete, serves to indicate the great variety of uses to which glycerin is applied.

CHAPTER XV.

EXAMINATION OF RAW MATERIALS AND FACTORY PRODUCTS.

Examination of Raw Materials and Factory Products. Soda-ash. Determination of Total Alkali. Commercial Caustic Soda. Determination of Total Alkali. Determination of Sodium Hydrate. Determination of Sodium Chloride. Soap-stock. Determination of Moisture. Determination of Free Fatty Acids. Color and Odor. Melting-point. Alkali Absorption. Unsaponifiable Matter. Titre Test. Melting-point of Fatty Acids. Rosin. Waste Soap Lye. Determination of Total Alkali. Determination of Free and Combined Alkali. Determination of Salt. Determination of Glycerin. Bichromate-oxidation Method. Acetin Method. Examination of Products Obtained in the Recovery of Glycerin. Alkalinity of Glycerin "Foots." To Test Recovered Salt for Sodium Chloride. Determination of Sodium Chloride in Crude Glycerin. Refined Glycerin. Specific Gravity. Salt. Ash. Carbonaceous Residue. Acidity. Color. Characteristics of Dynamite-refined Glycerin. Nitrating Test. Determination of Specific Gravity by the Hydrometer. Examination of Soap. Moisture. Mixed Fatty Acids. Total Alkali. Free Alkali. Alkaline Residue Insoluble in Alcohol. Silica. Insoluble Matter. Rosin. Fatty Acids. Determination of Iodin Absorption. Glycerin in Soap. Chlorides and Sulphates. Statement of Results.

Examination of Raw Materials and Factory Products.—The chemical and physical examination of the raw materials, of the various products obtained in the course of manufacture, and of soaps which it is desired to duplicate or for any reason to learn their composition, constitutes the general work of the factory chemist engaged in the soap-industry. While it by no means is intended to present an exhaustive examination of the analytical methods commonly employed for this purpose, which would be within the province of a work on chemical analysis, of which there are numerous treatises, it is entirely within the scope of the present work to describe the common tests and methods of examination employed in a soap-factory laboratory. Many of these tests are used in all factories that make any pretense to technical scrutiny of factory work; many are omitted as non-essential; while in many factories no attention whatever is paid to chemical analysis. Where the last condition prevails small-scale production is the rule. A chemical laboratory and competent direction therein are now recognized as an indispensable adjunct of every well-equipped plant. In the following sections it is proposed to present the more common tests that are used in the examination of the raw materials and factory products of soap-manufacture.

Soda-ash. Determination of Total Alkali.—The examination of soda-ash comprises chiefly the determination of sodium carbonate to ascertain the grade purchased. It may also be examined for sodium chloride, which, as has already been seen, exists in considerable amount in low-grade ash. In the chemical analysis of the various products to be enumerated it is presumed that the different standard solutions, such as semi-normal sulphuric acid, semi-normal caustic soda, tenth-normal silver nitrate, etc., are prepared and kept in a state of accuracy. Procedure: Weigh 1 to 2 grams of soda-ash into a 500-cubic-centimeter flask and add about 200 cubic centimeters of distilled water and a few drops of phenolphthalein as indicator. Run in from a burette an excess of semi-normal sulphuric acid and boil to expel all traces of carbonic acid gas. Titrate the excess of standard acid added with semi-normal caustic soda. The value of either solution is known in terms of the other and in terms of the various compounds of the caustic alkalis. The weight of sodium carbonate corresponding to the number of cubic centimeters of semi normal sulphuric acid required to neutralize it is divided by the weight of soda-ash taken, which gives the percentage of actual anhydrous sodium carbonate in the sample of soda-ash.

Commercial Caustic Soda. Determination of Total Alkali.—Weigh 3 to 5 grams of the sample into a 100-cubic-centimeter graduated flask; dissolve in distilled water and make the cooled solution up to the mark with additional water. Titrate an aliquot portion, say 10 cubic centimeters, proceeding as described in the determination of the total alkalinity of soda-ash. Express the percentage of total alkali in terms of sodium oxide Na_2O.

Determination of Sodium Hydrate.—Weigh out 1 to 2 grams of the sample in a 100-cubic-centimeter flask and dissolve in a convenient amount of distilled water. Precipitate with hot barium chloride (10 per cent aqueous) solution. Cool the solution and make up to the mark with distilled water. Allow the flask to stand until the precipitate of barium carbonate has subsided thoroughly. Filter the supernatant liquid and titrate any convenient number of cubic centimeters, say 10, with semi-normal hydrochloric acid. Standard sulphuric acid cannot be used as barium sulphate is insoluble. Calculate the number of cubic centimeters of standard hydrochloric acid used in terms of sodium hydrate and divide by the weight of the sample taken. Express the percentage as sodium hydrate in terms of sodium oxide.

Determination of Sodium Chloride.—Dissolve a weighed portion of the sample, 2 to 3 grams, in an accurately measured volume of distilled water and transfer 10 cubic centimeters to a porcelain dish 4 inches in diameter. Dilute the 10 cubic centimeters to a convenient volume with distilled water and neutralize, using dilute nitric acid and sodium carbonate solution, with phenolphthalein as indicator. When neutral add a few drops of potassium chromate as indicator and titrate to the appearance of a permanent reddish coloration with tenth-normal silver nitrate solution. Each cubic centimeter of the silver nitrate solution will contain 0.017 grams of silver nitrate which will precipitate 0.0493 grams of sodium chloride in accordance with the following proportion:

$$\underset{170}{AgNO_3} : \underset{58.5}{NaCl} :: 0.017 : x; \quad x = 0.04927.$$

Multiply the number of cubic centimeters of silver nitrate solution used by its value in sodium chloride and the result by the aliquot part of the whole solution used; divide the result by the weight of the sample taken which will give the percentage of sodium chloride.

The results of the determinations may be summarized as follows, taking an actual analysis as an example:

Total alkali as Na_2O	69.55	per cent
Sodium hydrate as Na_2O	61.72	" "
Combined alkali as Na_2O (by difference)	7.83	" "
Sodium chloride	9.40	" "

To express the complete results in a final form it is necessary to transform the sodium hydrate as Na_2O into sodium hydrate as $NaOH$, and the combined alkali as Na_2O into combined alkali as Na_2CO_3, which done we have the following report:

Total alkali, Na_2O	69.55	per cent
Caustic alkali, $NaOH$	79.64	" "
Carbonated alkali, Na_2CO_3	10.92	" "
Sodium chloride, $NaCl$	9.40	" "
Undetermined	——	

Judging from the above report this caustic soda is of practically 70° quality.

Soap-stock.—Samples of animal-stock are as a rule collected at the bung of the container by means of a sampler which may be inserted into the contents in various directions to observe any variation in quality. A sample is commonly taken from each barrel of the lot and the whole well mixed to insure uniformity of composition. If the individual samples differ greatly in quality it may be necessary to heat the mass to incipient fusion to insure uniformity. Samples of liquid-stock may be collected by means of a lye sampler, which consists of a tube, one end of which may be closed or opened by means of a ball attached to a wire and controlled at the opposite end.

Determination of Moisture.—For this purpose a hot closet maintained at a constant temperature of 105° C. by means of steam or other agent is necessary. From 3 to 5 grams of the well-mixed sample are weighed into a tared, shallow porcelain dish (No. 11 *6173; Eimer and Amend's Catalogue). The weighed sample is placed in the hot closet and maintained at a temperature of 105° C. for one hour, after which it is removed, cooled and weighed. The loss in weight divided by the weight of the sample taken gives the percentage of moisture present. With stock characterized by the presence of fatty acids of low molecular weight, such as cocoanut and palm-kernel oils, care should be taken to see that the loss in weight does not arise from their volatilization. With stock containing unsaturated fatty acids, such as cottonseed-oil, which is as a rule practically free from water, the sample may increase in weight from oxidation. With animal-stock the presence of moisture is frequently considerable and an allowance for same is commonly made when present over an agreed amount.

Determination of Free Fatty Acids.—Aside from the titre test and the observation of color and odor the determination of the free acidity is the most important test applied to soap-stock. As has been already discussed, it indicates the value of the stock as a source of glycerin as well as the origin of the stock and the care used in rendering. Owing to the greater activity of decomposition in summer than in winter, a higher percentage of free fatty acid may be expected during the former season, as is shown in the following table of determinations made on the stocks mentioned during the respective seasons:

Soap-stock.	Per Cent of Free Fatty Acids.	
	Summer.	Winter.
Tallow	7.85	5.85
Bone-stock	10.68	6.64
Grease	18.03	7.49

In the procedure of the determination the sample is heated to fusion and 5 cubic centimeters are transferred to a 250-cubic-centimeter wide-mouthed Erlenmeyer flask. Add about 50 cubic centimeters of 95 per cent neutral alcohol and insert in the mouth of the flask by means of a cork a long glass tube to serve as an air return-flow condenser. Heat the flask on the steam bath for five minutes. The determination is based upon the practical insolubility of neutral glycerides in alcohol in which the free fatty acids dissolve readily. When solution is complete remove the flask from the steam-bath and detach the condenser. Add a few drops of phenolphthalein as indicator and titrate with semi-normal caustic soda. To ascertain the percentage of free fatty acids multiply the number of cubic centimeters of semi-normal caustic soda required for neutralization by the value of 1 cubic centimeter in terms of oleic acid and divide the result by the weight of stock used.

Color and Odor.—The color and odor of any given sample are important indices of its quality and should never be neglected. Observation of these qualities, combined with that of the body and grain, comprise the sole tests applied to the greater part of all animal-stock bought and sold. Marked odor and color are generally associated with high free acidity. The body or firmness of the stock corresponding to its titre indicates its quality to carry rosin, and hence the yield of soap. The grain especially noticeable in warm weather indicates roughly the relative proportions of solid and liquid glycerides. A careful study of these properties is indispensable to every careful buyer of stock.

Melting-point.—The term "melting-point," as usually employed, means the temperature at which a globule of fat taken from the sample becomes transparent. This temperature, however, varies under different conditions, depending chiefly upon the initial temperature of the fat-disc, the time during which the temperature of the disc of fat is raised until it is globular and transparent and the consistency of the fat. The melting-point is considered to be that temperature at which a thin disc of fat, when freed from the attraction of gravitation, assumes a practically spherical state. Where a large number of determinations are made daily the apparatus devised by Wiley is arranged so that one attendant can conduct at least six determinations at once, the air required for agitating the water-bath being supplied by a small air-pump.

Wiley's method may be described as follows:

Preparation of Reagents.—(*a*) A piece of ice floating in distilled water that has been recently boiled.

(*b*) A mixture of alcohol and water of the same specific gravity as the fat to be examined. This is prepared by boiling distilled water and 95 per cent alcohol for ten minutes to remove the gases which they may hold in solution. While still hot the water is poured into the test-tube described below until it is nearly half full. The test-tube is nearly filled with the hot alcohol, which is carefully poured down the side of the inclined tube to avoid too much mixing. If the alcohol is not added until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disc of fat as the temperature rises and finally force it to the top.

Apparatus.—The apparatus for determining the melting-point consists of an accurate thermometer reading easily tenths of a degree; a cathetometer for reading the thermometer (but this may be done with an eye-glass if held steadily and properly adjusted); a thermometer; a tall beaker 35 centimeters high and 10 centimeters in diameter; a test-tube 30 centimeters long and 3.5 centimeters in diameter; a stand for supporting the apparatus; some method of stirring the water in the beaker (for example, a blowing bulb of rubber and a bent glass-tube extending to near the bottom of the beaker). (See Fig. 223.)

Determination.—The discs of fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of from 15 to 20 centimeters on a smooth piece of ice floating in distilled water that has been recently boiled. The discs thus formed are from 1 to 1.5 centimeters in diameter and weight about 200 miligrams. By pressing the ice under the water the discs are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice-water before using.

The discs must be allowed to stand for two or three hours, in order to obtain the normal melting-point.

The test-tube containing the alcohol and water is placed in a tall beaker containing water and ice until cold. The disc of fat is then dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test-tube and lowered until the bulb is just above the disc. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disc, the thermometer is moved from time to time in a circularly pendulous manner.

The disc having been placed in position, the water in the beaker is slowly heated and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° below the melting-point, the disc of fat begins to shrivel and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotatory movement should be given to the thermometer bulb. The rise of temperature should be so regulated that the last 2° of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so the reading of the thermometer is to be made. As soon as the temperature is taken the test-tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test-tube (ice-water

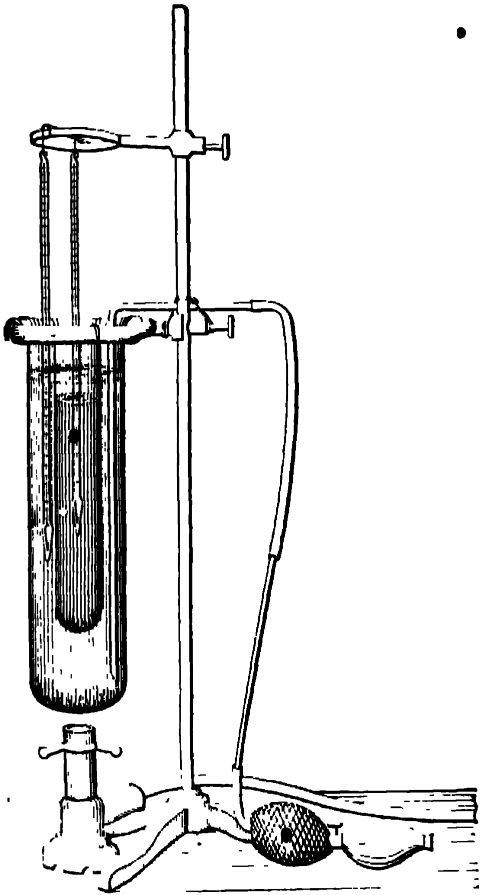


FIG. 223.—Apparatus for the Determination of the Melting-point.

having been used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum of about 1.5° above the melting-point of the fat under examination.

The edge of the disc should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place and the disc adhere to the sides of the tube a new trial should be made.

Alkali Absorption.—Weigh out 2 to 3 grams of the well-mixed sample into a 250-cubic-centimeter wide-mouthed Erlenmeyer flask. Add from a burette an accurately measured quantity of alcoholic potash solution sufficient to saponify the weight of fat used and to leave some excess. Insert the air condenser used in the free fatty acid determination and place the apparatus on the steam-bath. Heat with occasional shaking until assured of complete saponification, which ensues rapidly and is indicated by a clear solution. Remove the condenser, add a few drops of phenolphthalein as indicator and titrate the excess of caustic potash with semi-normal sulphuric acid. Ascertain the value of the alcoholic potash solution (25 grams KOH per litre of distilled water) in terms of the semi-normal sulphuric acid solution by titration. Transform the amount of alcoholic potash solution used into its equivalent of semi-normal sulphuric acid. From this figure subtract the number of cubic centimeters of semi-normal sulphuric acid used to neutralize the excess of potash. The result is the amount of alcoholic potash required to saponify the stock in terms of semi-normal sulphuric acid. This can be expressed directly in grams of NaOH or KOH, which figure divided by the weight of the sample taken will give the percentage of caustic alkali absorbed. If the percentage of caustic alkali absorbed is below the amount usually absorbed by stock of the general quality examined, the nature and amount of the inert or unsaponifiable matter must be determined.

Unsaponifiable Matter.—This determination may be made either (*a*) with a fresh sample or may be made a (*b*) continuation of the determination of the percentage of caustic alkali absorbed. By the former method (*a*) saponify 5 grams of oil or fat with alcoholic potash and remove the alcohol by evaporation. Wash into separatory funnel with from 70 to 100 cubic centimeters of water and extract with from 50 to 60 cubic centimeters of ether. In case the two liquids do not separate a few cubic centimeters of alcohol may be added. Separate the water solution and wash the ether with water containing a few drops of sodium hydrate. Again extract the soap solution and washings with ether and evaporate the combined extracts to dryness. In most cases it is advisable to add a little alcoholic potash to the residue and heat in order to saponify any traces of fats left unsaponified and extract again with ether. Transfer to a weighed dish and dry as quickly as possible in a water oven.

Many of the hydrocarbon oils are volatile at 100° C., so that the drying should not be carried any further than necessary.

On account of the solubility of soap in ether and petroleum ether it is well to wash the residue with warm water containing a little phenolphthalein. If it shows alkaline reaction there is soap present.

By the latter method (*b*) evaporate the contents of the Erlenmeyer flask to dryness. Break the residue into small particles with a glass-rod to facilitate extraction with petroleum ether which is then added. The air-condenser is then inserted and the residue is extracted on the steam-bath. When assured of complete extraction remove the flask from the steam-bath and cool; then filter the extract into a tarred beaker and evaporate the filtrate to dryness. The residue thus obtained is the unsaponifiable matter. If lime soap is present this may be ascertained by treating a sample of the stock with dilute sulphuric acid, whereupon a precipitate of calcium sulphate is produced. The presence of lime may be confirmed by the ammonium oxalate test.

Foreign matter, animal tissue, etc., may be determined by extracting a weighed portion of the sample with petroleum ether, whereupon all the fatty matter dissolves. Filter the extract (through a tared filter) and wash the residue on the filter free from all traces of fat with petroleum ether. Upon drying and weighing the residue thus treated the percentage of foreign matter may be ascertained.

Titre Test.—The titre or maximum temperature reached during the process of crystallization of the fatty acids is an important test for the commercial valuation of fats. The method as recommended by Wolfbauer is as follows: 120 grams of the fat are melted in a beaker at a temperature but slightly above its melting-point, mixed with 45 cubic centimeters of caustic potash solution (1250 grams of caustic potash in 1 litre of water), and stirred until the fat is completely emulsified. It is then covered and kept at 100° C. for two hours, being occasionally stirred. A small portion is then tested by warming with alcohol (50 per cent) to ascertain whether saponification is complete, indicated by a clear solution; otherwise, it must be replaced in the bath and there allowed to remain until this is accomplished.

The soap is now decomposed by boiling with 165 cubic centimeters of dilute sulphuric acid (sp. gr. = 1.142 = 18° Bé.), preferably done in a silver dish, and continued until the free fatty acid rises to the top as a perfectly oily layer. The silver dish is then covered with an evaporating dish filled with cold water to check the evaporation. The aqueous solution is then completely drawn off, and the fatty acid washed by boiling one-quarter of an hour with dilute sulphuric acid (5 cubic centimeters of concentrated sulphuric acid and 100 cubic centimeters of water). After settling and removing the dilute acid, it is boiled with 100 cubic centimeters of pure water, this last being repeated should the washings taste distinctly acid. It is then dried in an open dish at 100° C. for two hours.

Only fatty acids obtained as above can be considered sufficiently pure and dry to be used for the determination of the freezing-point.

In the determination proper the following apparatus is employed: A thin-walled test-tube, $3\frac{1}{2}$ centimeters by 15 centimeters is fixed by means of a cork in a suitable bottle. A Centigrade thermometer, extending from 1° to 60°, and graduated in fifths of a degree, is fixed in the test-tube by a second cork, which must be sufficiently loose to permit of an easy stirring of the contents of the tube with the thermometer. As the thermometer should be as short as possible, its scale is shortened by an enlargement blown in the bore in the interval between 2° and 28.° The amount of mercury *above* the surface of the fatty acid is thus diminished, and a very appreciable error (a lowering of the freezing-point) is consequently avoided.

To accomplish the determination the test-tube is filled to within 1 centimeter or 1½ centimeters of the top with the melted fatty acid, the thermometer immersed in the liquor to about the 35°, mark (when the instrument should clear the bottom of the tube by about 4 or 5 centimeters), and the liquid stirred until it becomes quite opaque, and partial solidification sets in. Care should be taken at this point that the thermometer be not more deeply immersed, and after stirring rapidly in a circle ten more times the thermometer is allowed to stand. The mercury now begins to rise in consequence of the latent heat liberated from the solidifying fatty acid; the highest temperature noted may be taken as the freezing-point.

The reading of the thermometer should be corrected for its inherent errors, previously determined. Its zero point should also be redetermined from time to time.

Each freezing-point determination should be repeated, and the difference between the two should not exceed 0.1° C.; as a rule, it will not exceed 0.05.

The use of narrower test-tubes than above prescribed, as well as lack of attention to other details, generally leads to low results as a so-called "over-cooling" always appears in the passage from the liquid to the solid state.

Relative to variations in the procedure it may be stated that saponification of the fat may be effected either by the use of an aqueous or an alcoholic potash solution. A variation in the length of time of saponifying does not influence the final result.

Regarding the minimum length of time necessary, it varies with different fats—slightly rancid fats requiring less than fresh fats, some of the former undergoing even spontaneous saponification on being mixed with the caustic potash. As a rule, however, for fresh fats two hours is amply sufficient, provided the fat was not melted at too high a temperature, and that, after adding the caustic potash, a perfect emulsion was obtained by a vigorous stirring.

Comparative tests have shown most conclusively that by increasing the amount of water in a fatty acid the freezing-point is lowered, and *vice versa.* Further, as the absorbent power of fatty acids for water is less in the cold than at a higher temperature, it follows that during the cooling of a fatty acid, saturated with the maximum amount of water, at a certain temperature a separation of water must take place, shown by the still liquid fatty acid becoming turbid. This turbidity naturally interferes with the detection of solidification, and consequently determinations made on undried or partially dried fatty acids show much less agreement with each other than those made on the perfectly dried acid. In two determinations on the same fat, using the undried fatty acid, were obtained the following:

Freezing-points	43.14°
and	42.86°
Difference	0.28°

If, after washing a fatty acid, a filtration be substituted for the drying, a partial removal of the water is thus effected. But as the freezing-point of a fatty acid so treated is 0.3° to 0 4° lower than one similarly treated, but also dried, it appears that such a filtration is entirely superfluous.

It makes no difference in the final result whether the fatty acid be dried only one and one-half hours or a much longer time. It is also immaterial whether the dried fatty acid be used immediately for the determination, or whether it be first allowed to solidify and to stand a long or short time, being subsequently remelted for the determination.

Wolfbauer showed that the diameter of the tube may be increased from 3½ centimeters without disturbing the ratio between the amount of heat radiating from the fatty acid through the walls of the tube and the amount of liberated latent heat. Consequently a tube 3½ centimeters or more in diameter answers perfectly for the correct determination of the freezing-point, but it is not permissible to use one appreciably narrower than this.

The titer test as employed in the United States Department of Agriculture, Circular 22, Bureau of Chemistry, is carried out as follows:

Weigh 75 grams of fat into a metal dish and saponify by using 60 cubic centimeters of 30 per cent sodium hydrate (36° Bé. caustic soda) and 75 cubic centimeters of 95 per cent by volume alcohol, or 120 cubic centimeters of water. Boil down to dryness, with constant stirring to prevent scorching. This should be done over a very low flame, or over an iron or asbestos plate. Dissolve the dry soap in a liter of boiling water, and if alcohol has been used boil for 40 minutes in order to remove it, adding sufficient water to replace that lost in boiling. Add 100 cubic centimeters of 30 per cent sulphuric acid (25° Bé. sulphuric acid) to free the fatty acids and boil until they form a clear transparent layer. Collect the fatty acids in a small beaker and place on the steam-bath until the water has settled, then decant them into a dry beaker, filter, using a hot-water funnel, and dry 20 minutes at 100° C. When dried, cool the fatty acids to 15–20° C. above the expected titer and transfer to the titer-tube, which is 25 millimeters in diameter and 100 millimeters in length (1 in. by 4 in.) and made of glass about 1 millimeter in thickness. This is placed in a 16-oz. salt-mouth bottle of clear glass, about 70 millimeters in diameter and 150 millimeters high ($2\frac{4}{5}$ in. by 6 in.), fitted with a cork, which is perforated so as to hold the tube rigidly when in position. The thermometer, graduated to 0.1° C., is suspended so that it can be used as a stirrer, and the mass is stirred slowly until the mercury remains stationary for 30 seconds. The thermometer is then allowed to hang quietly, with the bulb in the center of the mass, and the rise of the mercury observed. The highest point to which it rises is taken as the titer of the fatty acids.

The fatty acids are tested for complete saponification as follows:

Three cubic centimeters of the fatty acids are placed in a test-tube and 15 cubic centimeters of alcohol (95 per cent by volume) added. The mixture is brought to a boil and an equal volume of ammonia (0.96 sp. gr.) added. A clear solution should result, turbidity indicating unsaponified fat. The room temperature must be reported.

Melting-point of Fatty Acids.—The melting-point of the fatty acids cannot be determined in the same apparatus and by the same methods as those described for the fats themselves because the acids are soluble in alcohol. It should be remembered that the melting-point of the fatty acids is slightly above that of the glycerides. The determination is made by drawing up the melted fatty acid into a very thin-walled capillary tube 1 or 2 inches long, according to the length of bulb of the thermometer used. Seal one end of the tube and allow the fatty acid to cool on ice for from 12 to 15 hours. Then attach to the bulb of a delicate thermometer graduated to one-fifth degree, immerse in a beaker of water, and warm up very slowly. The point where the acid becomes transparent is taken as the melting-point.

Rosin.—The chemical test usually applied to rosin is the determination of the unsaponifiable matter which increases as the quality of the rosin diminishes. The physical grading accorded the barrel from which the sample is taken is made by comparing the depth of color of a cube, cut from the sample, with dimensions equal to those of a standard cube, with the depth of color of sample standard cubes. Rosin of a given grade should correspond in color and clearness to the color and clearness of the standard cube of the same grade. Standard cubes should be renewed from time to time as they tend to bleach under exposure and to lose the cube shape by softening. In comparing rosin samples with the rosin standards strict uniformity of dimension looked through and exposure to light must be observed.

Waste Soap Lye.—The examination of waste soap lye comprises the volumetric tests most commonly made in the soap laboratory, viz., total alkali, both free and combined, salt and glycerin. Where these tests are numerous many may be carried on together and finished successively. The chemical and physical characteristics of waste lye have already been studied.' While the tests and procedures presented in this section lay no claim to the accuracy of refined analysis, they are distinctively factory tests and find their greatest recommendation in the combined speed and accuracy with which they may be made.

Determination of Total Alkali.—Transfer by means of a pipette to a 500-cubic-centimeter flask 10 cubic centimeters of the sample free from soap and foreign matter. Add about 150 cubic centimeters of distilled water; add phenolphthalein as indicator; run in from a burette sufficient semi-normal sulphuric acid to permanently discharge the pink coloration produced by the indicator; boil to expel all traces of carbonic acid gas (5 minutes boiling will suffice) and titrate the excess of standard acid used with semi-normal caustic soda. To ascertain the percentage of total alkali expressed as NaOH by volume multiply the number of cubic centimeters of $\frac{N}{2}H_2SO_4$ absorbed by 0.2. The percentage by weight would be slightly less.

Determination of Free and Combined Alkali.—To determine the relative proportions of free and combined alkali in the total alkali proceed as outlined for the determination of the corresponding bodies in commercial caustic soda.

Determination of Salt.—Transfer by means of a pipette 5 cubic centimeters of the sample to a 100-cubic-centimeter beaker and add 50 cubic centimeters of distilled water. Mix thoroughly and transfer 5 cubic centimeters of the diluted waste lye to a 4-inch porcelain evaporating dish. Neutralize the solution as already described in the same determination in commercial caustic soda. Likewise titrate with $\frac{N}{2}AgNO_3$ solution (using potassium chromate as indicator) and calculate the percentage of sodium chloride as though 0.5 gram of lye had been taken for analysis.

Determination of Glycerin.—The analytical methods employed industrially for the determination of glycerin are based upon the complete oxidation of the glycerin to carbon dioxide and water. Potassium permanganate in either acid or alkaline solution may be used according to two methods as the oxidizing agent. In the more common and, as a rule, the more satisfactory oxidation method, potassium bichromate and sulphuric acid are used. In the acetin method the quantitative determinatian of glycerin is based upon its transformation into glyceryl tri-acetate. The great source of error in oxidation methods is the liability of oxidizing organic matter as well as glycerin. Oxidation methods give uniformly high results. In the acetin method the glycerin alone is susceptible to the action of acetic anhydride. The method, however, is time-consuming; it is not applicable to waste lye and other products low in glycerin owing to the hydrolysis of the unstable glyceryl tri-acetate in the presence of water; it gives results too low, and is not well adapted as an industrial laboratory method. Its chief value lies in its use as a check upon the more rapid and more easily manipulated oxidation methods.

Bichromate-oxidation Method.—The glycerin is oxidized as stated by the use of potassium bichromate and sulphuric acid and the excess of the bichromate is titrated with ferrous ammonium sulphate. The reaction takes place in accordance with the following equation:

$$3C_3H_8O_3+7K_2Cr_2O_7+28H_2SO_4$$
$$=7K_2SO_4+7Cr_2(SO_4)_3+40OH_2OH_2+9CO_2$$

Strength of Solutions.—Potassium bichromate: 75 grams of $K_2Cr_2O_7$ and 150 grams of H_2SO_4 (sp. g. 1.84) per litre. Ferrous ammonium sulphate: 300 grams of $FeNH_4(SO_4)_2$ and 10 cubic centimeters H_2SO_4 (sp. g. 1.84) per litre. Standardizations: To standardize the ferrous ammonium sulphate solution run out 7 to 10 cubic centimeters of the bichromate solution into a 250-cubic-centimeter beaker; add 15 cubic centimeters of (1.84) sulphuric acid and heat for one-half hour on the steam-bath; dilute to 200 cubic centimeters and titrate. To standardize the bichromate solution against glycerin, weigh out 0.05 gram of chemically pure glycerin into a 250-cubic-centimeter beaker; add about 15 cubic centimeters of bichromate solution and then slowly 15 cubic centimeters of (1.84) sulphuric acid; heat for one-half hour on the steam-bath and titrate the excess of bichromate with ferrous ammonium sulphate solution. In titrations of the bichromate and ferrous ammonium sulphate solutions potassium ferricyanide is used as an indicator with a porcelain tile.

Procedure.—Weigh out 1.5 to 2 grams of the sample into a 25-cubic-centimeter graduated flask and add 1.5 grams of moist silver sulphate, weighed on the rough laboratory balance. The solution is made up to the mark with distilled water and 6 drops of water are added to displace the volume of the precipitant added. The contents of the flask are then shaken until all the sodium chloride has been precipitated, indicated by the solution being perfectly clear;

after which the supernatant liquid is filtered. A portion of the filtrate is used to rinse the 5-cubic-centimeter pipette. Five cubic centimeters are now pipetted into a 250-cubic-centimeter beaker. Bichromate solution, 7 to 10 cubic centimeters, is now run in, after which 15 cubic centimeters of (1.84) sulphuric acid are added. The contents of the beaker (at all times covered with a watch-glass) are heated for one-half hour on the steam-bath. The solution is then diluted to about 200 cubic centimeters and the excess of bichromate is titrated with ferrous ammonium sulphate solution. The utmost uniformity of procedure should be followed, especially as to the excess of bichromate and to the degree of dilution.

Acetin Method.—For determination of glycerin in crude glycerin the acetin method finds its true province. It depends on the phenomenon that glycerin on boiling with acetic anhydride is quantitatively transformed into triacetin. If the latter be then dissolved in water and the free acetic acid be neutralized with sodium hydrate, the dissolved triacetin can be saponified with caustic soda and the excess of the latter titrated back.

The necessary reagents are:

(1) Semi-normal to normal hydrochloric acid accurately standardized.

(2) Dilute caustic soda, not standardized, which contains not more than 20 grams sodium hydrate to the litre.

(3) Concentrated, about 10 per cent, caustic soda, best preserved in a flask provided with a 25-cubic-centimeter pipette. 1 to 1.5 grams of the sample are weighed out in a wide-necked, small, round-bottom flask of about 100 cubic centimeter capacity. About 8 grams acetic anhydride are added, with about 3 grams of dehydrated acetate of soda.

The anhydrous salt is prepared by heating sodium acetate to complete dryness in the hot-closet. A reflux condenser is attached and the contents of the flask are boiled for 1½ hours, whereupon they are cooled and 50 cubic centimeters of warm water are added through the reflux condenser. Agitate the flask to affect solution of the triacetin but do not boil. When solution is complete filter the liquid from the flocculent precipitate which constitutes most of the impurities. contained in the sample. Make the filtrate up to a convenient volume and neutralize the free acetic acid in an aliquot portion with an 8 to 10 per cent solution of carbonic acid-free caustic soda, finishing for the sake of accuracy with a more dilute solution, using phenolphthalein as indicator.

The amount of acetic anhydride that has combined with the glycerin can now be determined by simple saponification: Add 50 cubic centimeters semi-normal caustic soda to the contents of the flask and boil 15 minutes. Titrate back with semi-normal acid. Each cubic centimeter semi-normal alkali used is equivalent to 0.01533 grams glycerin.

Examination of Products Obtained in the Recovery of Glycerin.—The analytical work connected with the recovery of glycerin from waste soap lye comprises chiefly the examination of waste lye for total alkalinity preparatory to calculating the amount of sulphuric acid and other chemicals used to clarify it; the determination of the alkalinity of glycerin "foots" and the amount of sulphuric acid required to neutralize it. Half-crude, or the product of the first stage of concentration of waste lye, may be examined for glycerin and sodium chloride. Crude glycerin is usually examined for sodium chloride, ash, and glycerin. The product of the first distillation may be examined for acidity to ascertain the amount of soda-ash required to neutralize it. The product of the second distillation, dynamite-refined glycerin, is usually examined for ash, sodium chloride, carbonaceous residue, acidity, both free and combined, specific gravity and color. The tests already given combined with those applied to glycerin of dynamite-refined grade comprise the more common ones.

Alkalinity of Glycerin "Foots."—Weigh 5 grams of the sample into a platinum dish. Exaporate to dryness, then heat to ignition, using potassium chlorate to remove all carbonaceous matter and to transform sulphides to sulphates. Digest the residue and transfer it to a 500-cubic-centimeter flask. Determine the total alkali as before described and express the result in terms of NaOH. Therefrom the volume of sulphuric acid required for neutralization may be readily calculated.

To Test Recovered Salt for Sodium Chloride.—Weigh out 2 grams of ignited salt on the rough balance, and dissolve in 200 cubic centimeters of distilled water. Take 5 cubic centimeters and titrate in the manner as before described with $\frac{N}{10}$ $AgNO_3$ solution. Calculate as though 0.05 grams had been taken for analysis

Determination of Sodium Chloride in Crude Glycerin.—Weigh about 2.5 grams of the sample into a 25-cubic-centimeter graduated flask and make up to the mark with distilled water. Pipette 5 cubic centimeters into a beaker and dilute with 50 cubic centimeters of distilled water. Add 2 drops of phenolphthalein as indicator, and neutralize if necessary with nitric acid. Add 15 drops of indicator using potassium chromate (30 grams per litre) and titrate the solution with $\frac{N}{10}$ $AgNO_3$. The $AgNO_3$ solution is standardized against chemically pure salt.

Refined Glycerin.—Specific Gravity.—The Westphal balance is commonly employed for determining the specific gravity, although the specific gravity bottle may be used.

The Westphal balance is constructed with a plummet weighing 5 grams and occupying the space of 5 cubic centimeters. This is called Reimann's patent thermometer-body. The beam is graduated into ten equal divisions, and the heaviest weight weighs 5 grams. The next size weight weighs a tenth of this. The next, a hundredth, and the smallest a thousandth. The balance being adjusted in air, so that the pointers are exactly opposite, if the plummet is immersed in distilled water at 15° C., the heaviest weight being placed on the hook with the plummet should bring the pointers opposite each other again. When using the Westphal balance—applicable to all liquids except such as are too viscous, but works best with transparent liquids—set up the balance in a level place by placing the beam in position and hanging the plummet on the hook provided for it. Now adjust with the adjusting-screw in the foot until the pointers are exactly opposite each other; cool the liquid to be tested to a point a little lower than that at which the balance was graduated; fill the jar with enough of the liquid to a little more than cover the plummet and place the jar in position. Allow the temperature of the liquid to rise with occasional stirring, until the reading of the thermometer in the plummet is that at which the balance was graduated. Now hang the weights on the beam at the notches provided for them, until the pointers are again exactly opposite each other. If two belong on the same notch, hang the second on the hook of the first. Read the marks on the beam where the weights hang, and set down the figures by the side of each other, putting the figure under the heaviest weight at the left hand, that under the next heaviest, next, and so on. The result will be the specific gravity expressed in decimals

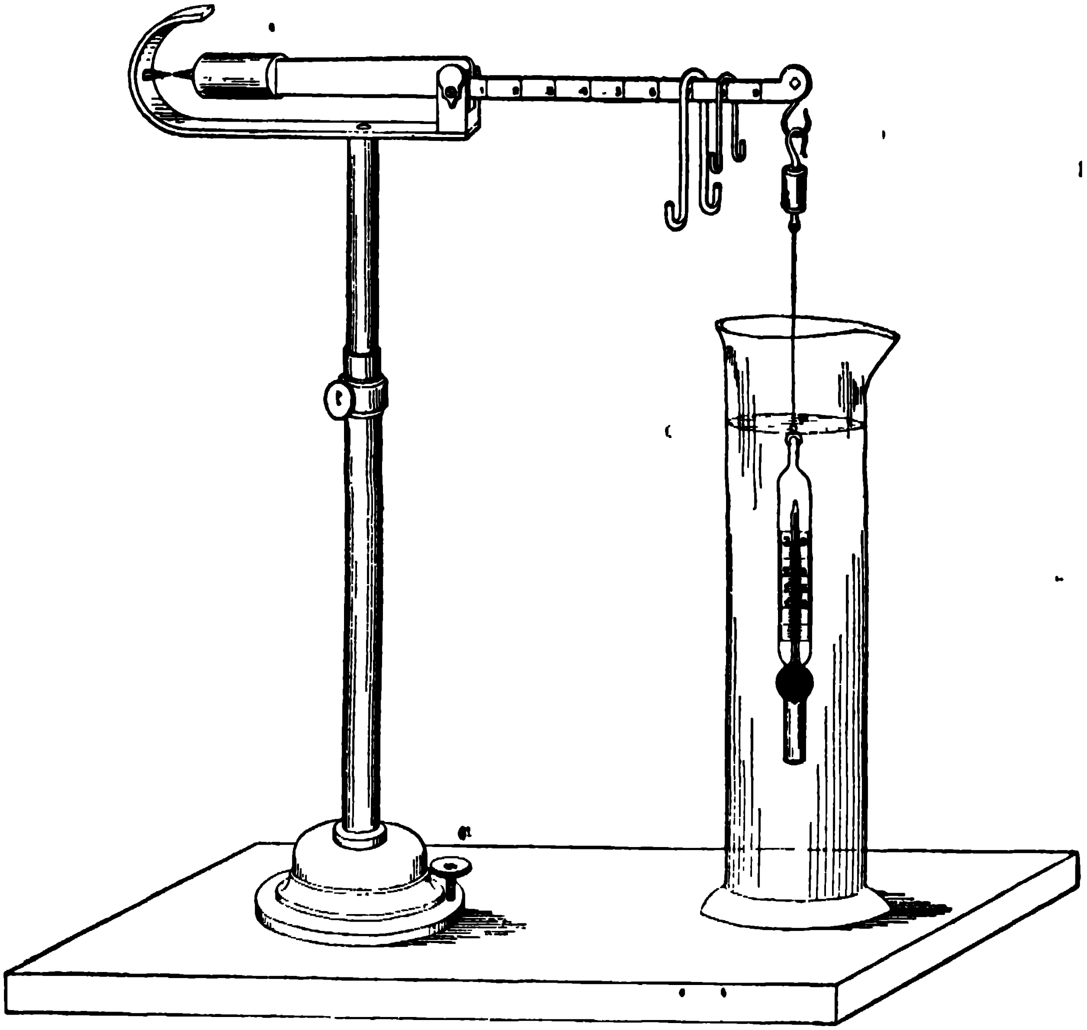


FIG. 224.—The Westphal Balance.

when the liquid is lighter than water. For liquids heavier than water it will be found that one of the heaviest weights will hang on the hook along with the plummet, the others being distributed along the beam. The reading of the heaviest weight will then be 1, which must be followed by a decimal point, the other readings following the order of the size of the weights, as above described, being placed after this decimal point. In case the specific gravity of the liquid in question is 2 or above, or 3 or above, two or three of the heaviest weights, as the case may be, will hang on the hook, the others being distributed along the beam. When the liquid to be tested is opaque, so that the reading of the thermometer in the plummet cannot be made, it is essential to have an additional thermometer to use in determining the temperature. This additional thermometer should, of course, be compared with the one in the plummet.

With all liquids which are transferred from one vessel to another there is always possibility of air being mechanically carried along with the liquid. While it is necessary to expel this air by heating the sample, it must be cooled again before the specific gravity is taken. Under no consideration should bubbles of air be allowed to adhere to the plummet. Allowing the liquid to stand for some time in the jar serves to free it from bubbles. In the standardization of the balance and in the determination of the specific gravity of glycerin, the temperature basis is, as a rule, that of 15° C. or 60° Fahr. The thermometer outside the liquid should not vary more than 5° from the plummet thermometer. Within this range of temperature, the factor of 0.0005 for each degree centigrade above or below 15° C. is commonly used for reducing the temperature of the determination to that of the standard. If above 15° C. the result is to be added to the observed specific gravity; if below 15° C. the result is to be subtracted.

Salt.—Weigh out 50 grams of the sample into a tared nickel dish and allow the glycerin to burn after heating carefully to the point of ignition. Burn off carbonaceous matter without loss of salt. Digest the residue with hot water and transfer to a porcelain dish and titrate with $\frac{N}{10}$ $AgNO_3$ solution.

Ash.—Weigh out 25 grams of the sample into a tared paltinum dish and heat carefully until the glycerin will ignite. Allow the glycerin to burn until it is consumed; then heat carefully with a Bunsen burner until all the carbonaceous matter is driven off, taking care in the operation that no salt is lost by decrepitation. Cool the platinum dish and residue in a dryer and weigh.

Carbonaceous Residue.—Weigh accurately 5 grams of the sample into a tared porcelain crucible. Heat to ignition in a hood, or place free from drafts and allow the glycerin to burn. After which flash the crucible with the flame of the Bunsen burner to expel adhering traces of glycerin without burning the non-glycerin matter Care must be exercised in this test to observe the strictest uniformity. of procedure.

Acidity.—The free acidity of refined glycerin may be attributed chiefly to fatty acids that have escaped removal in the process of distillation. They persist in well-made glycerin in very minute quantities. Owing to the uncertainty of their exact nature, their amount is expressed usually in the percentage of sodium oxide required to neutralize them. By combined acidity is meant the glyceride which results from the combination of the free acid bodies with glycerin. In the determination of free and combined acidity 10 grams of the sample are dissolved in 200 cubic centimeters of perfectly neutral water; add phenolphthalein as indicator. Find the exact volume of $\frac{N}{2}$ caustic soda solution required to effect neutralization in the cold. This volume, expressed as Na_2O, gives the free acidity. To determine the combined acidity add 1 cubic centimeter of $\frac{N}{2}$ caustic soda solution for every 5 grams of the sample taken. Boil for 5 minutes and titrate back with $\frac{N}{2}$ H_2SO_4. The new reading expresses the combined acidity.

Color.—Chemically pure glycerin is colorless and will remain unchanged on exposure. Glycerin of dynamite-refined grade tends to discolor on aging, which characteristic is due not to glycerin itself but to the contained impurities. The exact nature of the bodies undergoing change on aging has not been definitely ascertained, but they are supposed to be salts of fatty acids with heavy metals present in almost infinitesimal amount.

Characteristics of Dynamite-refined Glycerin.—The following characteristics of good dynamite-refined glycerin are given by Lewkowitsch:*

Specific gravity not lower than 1.2610 at 15° C. CaO, MgO, Al_2O_3 should be absent. Chlorin should be present in only a trifling amount. Arsenic must not be present in more than traces—Gutzeit's test is too severe.

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* Chem. Zeit., 1895, 19, 1423-24.

Gutzeit's test consists in allowing the gas, slowly liberated by the action of sulphuric acid on zinc in presence of the liquid supposed to contain arsenic, to come in contact with three thicknesses of filter-paper moistened with a saturated acid solution of silver nitrate. In order that sulphides which might be formed may not reach the filter-paper a plug of cotton or glass-wool, moistened with lead acetate solution, is introduced into the mouth of the test-tube. The presence of arsenic is shown by the production on the paper cap of a bright yellow stain which turns dark on the application of water. This test has been modified by substituting hydrochloric acid for sulphuric acid and doing away with the moistened plug of cotton; also by using a solution of mercuric chloride instead of silver nitrate. It suffices to make the sample just alkaline with NH_4OH and add $AgNO_3$; no yellow turbidity should appear. Organic impurities: 1 cubic centimeter of the sample diluted with 2 cubic centimeters of distilled water should give no coloration with a few drops of a 10 per cent $AgNO_3$ solution. Total solid residue should not exceed 0.15 per cent. Free acidity: Volatile fatty acids may be recognized by the ethereal smell produced by heating the sample with alcohol and sulphuric acid. In the following table are presented actual analyses of well-made dynamite-refined glycerin:

ANALYSES OF DYNAMITE-REFINED GLYCERIN.

Salt.	Ash.	Carbonaceous Residue. Per Cent.	Acidity.		Specific Gravity at 15° C.
			Free. Per Cent.	Combined. Per Cent.	
0.003	0.0032	0.04	0.0042	0.045	1.2641
0.001	0.001	0.012	0.0058	0.025	1.2632
0.001	0.002	0.018	......	0.022	1.2642
0.001	0.002	0.014	......	0.04	1.2625

The specific gravity of American glycerin for dynamite purposes averages about 1.2615 at 15° C.

Nitrating Test.—A crucial test of the fitness of glycerin for dynamite making consists in actually carrying out the manufacture of nitro-glycerin on a laboratory scale. The nitrating test, as recommended by Lewkowitsch, is as follows: 375 grams of a mixture of sulphuric and nitric acids (1 part by weight of HNO_3, specific gravity 1.50, with 2 parts of H_2SO_4, specific gravity 1.845) are prepared and allowed to cool.

The exact quantity is weighed out into a beaker of 500 cubic centimeters capacity and the beaker is placed in a large vessel through which a stream of cold water passes. The rubber tube leading the water to the cooling vessel must be wired on to the tap lest it slip off and cause water to splash into the nitrating mixture. Such an accident might lead to an explosion. The beaker should be thin so that is may be quickly broken with the thermometer used as a stirrer should heating begin after mixing. When the temperature of the mixture is 12° to 15° C., 50 grams of the glycerin to be tested are allowed to fall drop by drop into the acid mixture while the liquid is well stirred with the thermometer and the temperature carefully observed. The operation must be carried out slowly and cautiously, no further glycerin being added until the temperature of the mixture is below 25° C. A temperature of 30° C. should never be exceeded. When all the glycerin has been added stirring is continued until the temperature has fallen to 15° C., when the mixture is transferred to a perfectly dry separatory funnel which may be previously washed out with strong sulphuric acid. If the glycerin be of good quality the nitro-glycerin separates quickly as an oily somewhat turbid layer. The quicker and sharper the separation the better the sample. Should flocculent matter appear in the nitro-glycerin, or should the separation not take place within 5 to 10 minutes, or should an intermediate layer persist between the two liquids, the glycerin is not fit for dynamite making. In a very poor sample the nitro-glycerin remains diffused throughout the acid liquid for hours. The test may be made quantitative by reweighing the vessel used to contain the 50 grams of glycerin, and by running off the acid liquid from beneath the nitro-glycerin and swirling the nitro-glycerin around within the separatory funnel to collect stray drops of acid which may then be drawn off; no shaking is permissible. The nitro-glycerin is washed with water at 35° to 40° C., then once or twice with 20 per cent sodium hydrate solution and again with water. The volume of nitro-glycerin is read off in a 100-cubic-centimeter burette and when multiplied by 1.6 gives the weight in grams. This method, though rough, is satisfactory for technical purposes. The yield of nitro-glycerin should be from 219 to 230 per cent, the theoretical yield being 246.7 per cent.

In an investigation to determine the influence of the specific gravity of glycerin upon the yield of nitro-glycerin, Garrigues found that the small quantities of water present in dynamite glycerin is without effect, and that at least above a specific gravity of 1.257, or 97 per cent pure, the comparative value of glycerin is exactly according to its percentage strength, provided the mixed acids are of good quality. The mixture employed had the following composition: Sulphuric acid, 67.0 per cent; nitric acid, 26.1 per cent; hyponitric acid, 1.9 per cent; water, 5.0 per cent. Garrigues conducted the nitration as follows:

A dropping bottle is filled to such a point with the sample of glycerin that it will have delivered about 25 grams when drained. This is weighed before and after the nitration, the difference being the glycerin used.

One hundred cubic centimeters of mixed acids, about 178 grams, are placed in a dry beaker 2½ inches wide by 4½ inches deep, which is suspended in a large porcelain dish by passing through a piece of stiff cardboard. The cardboard is cut out to fit tightly around the beaker and is sufficiently large to completely cover the dish. This arrangement serves the double purpose of holding the nitrating vessel firm, and effectually preventing the splashing of any water into the acids, which would obviously be dangerous.

Cold water is allowed to circulate around the beaker throughout the operation, ice being used if necessary. When the acids have cooled to 10° C., the addition of glycerin is begun, drop by drop, but at such a rate that the temperature rises to 20° C. in about 3 minutes, the mixture being meanwhile stirred constantly with a short thermometer. The further addition is so regulated that the temperature remains between 19° and 21° C. When all the glycerin is in and the mercury has fallen to 15° C., the beaker is removed from its improvised water-jacket, wiped dry, and its contents transferred to a dry separating funnel, the beaker being rinsed with strong (1.84) sulphuric acid. The separation will begin at once, if both glycerin and acids are what they should be, but it is best to allow the mixture to stand several hours, when it will be perfectly clear below the layer of nitro-glycerin.

The acid is now tapped off and the upper layer run into 200 cubic centimeters of water at about 15° C. Stir up well, decant and repeat. Finally wash through a funnel into a narrow measuring tube graduated in 1 to 10 cubic centimeters: let settle until the volume remains constant at 15° C., and take the reading, which multiplied by the specific gravity, 1.6, gives the weight of nitro-glycerin.

This is the neatest manner of performing the nitrating test in the laboratory, though much time may be saved by running the whole mixture of acids and nitro-glycerin, immediately after nitrating into two litres of cold water, stirring meanwhile, then washing and measuring as above. This does away with the long wait for the clear separation, it taking place at once on contact with the water.

A comparison of the two methods has a bearing on the relative merits of the so-called direct and indirect separation, or separation and drowning as it is frequently termed. In Germany the wasteful process of drowning seems to be obsolete, but in this country many explosive works continue to enrich the rivers with countless tons of nitric and sulphuric acids yearly, much to the comfort of the acid makers.

It is interesting to note that the drowning process invariably returns a slightly better yield than the separation, but from the fact that in the former case the nitro-glycerin is milky and opaque, as against opalescent to transparent in good separation, it is not unlikely that the apparent increased yield is due to contained water. The following comparative tests on the same sample of glycerin, nitrated with acids from the same drum, illustrate this difference:

Specific Gravity.	Per Cent Yield by Separation.	Per Cent Yield by Drowning.
1.2619	233.0	235.5
		237.0

It may be said, and justly, that the difference here is no greater than the variation of duplicates nitrated in the same manner. The figures are chosen from a large number of trials with many samples of glycerin, and represent fairly the average observed difference. When several nitrations are made in each way, the average yield from drowning has been invariably in excess of that obtained as the average of separating. It was also in every case easy to distinguish the product by appearance, as noted before.

Determination of Specific Gravity by the Hydrometer.—This is an indirect method of ascertaining the specific gravity of liquids. The instruments commonly employed for this purpose are made of glass. They consist of a bulb or float filled with air, a stem placed above this float and bearing a scale, and a smaller bulb, a counterpoise, placed beneath the float and weighted with mercury or shot, so as to keep the instrument in an upright position when placed in the liquid. A body immersed in a liquid floats when it has displaced its own weight of liquid. It is, therefore, evident that a hydrometer will sink more deeply in a liquid of less density than it will in a liquid of greater density.

Hydrometers are provided with scales which either indicate specific gravity values or else bear an arbitrary valuation. On the former instruments the spaces between successive degrees are unequal. These hydrometers are constructed on the principle that equal differences of specific gravities are indicated by quantities proportional to the differences of the reciprocals of the specific gravities. On the latter instruments, the divisions are usually equal. Different kinds of hydrometers with arbitrary scales are used in the industries.

To determine the specific gravity of a liquid by means of a hydrometer, due attention being paid to the temperature of the liquid, the instrument is placed in the liquid and that degree of the scale in contact with the surface of the liquid is noted. If the hydrometer is graduated according to specific gravity values, the figure thus indicated denotes the specific gravity of the liquid. If bearing an arbitrary scale, conversion unto the corresponding specific gravity values must be made by calculation, or else determined by the aid of tables, similar to the one appended, for this purpose.

Among the numerous hydrometers bearing arbitrary scales, those devised by Antoine Baumé in 1768, and bearing his name, are probably more extensively used than any other kind. Although Baumé described very accurately the manner in which he obtained the scales for his two instruments—the one for liquids heavier, the other for liquids lighter than water—yet in the course of time the makers of these instruments deviated from his directions and in consequence there resulted great confusion as to the actual relation between the values of the so-called Baumé degrees and specific gravity.

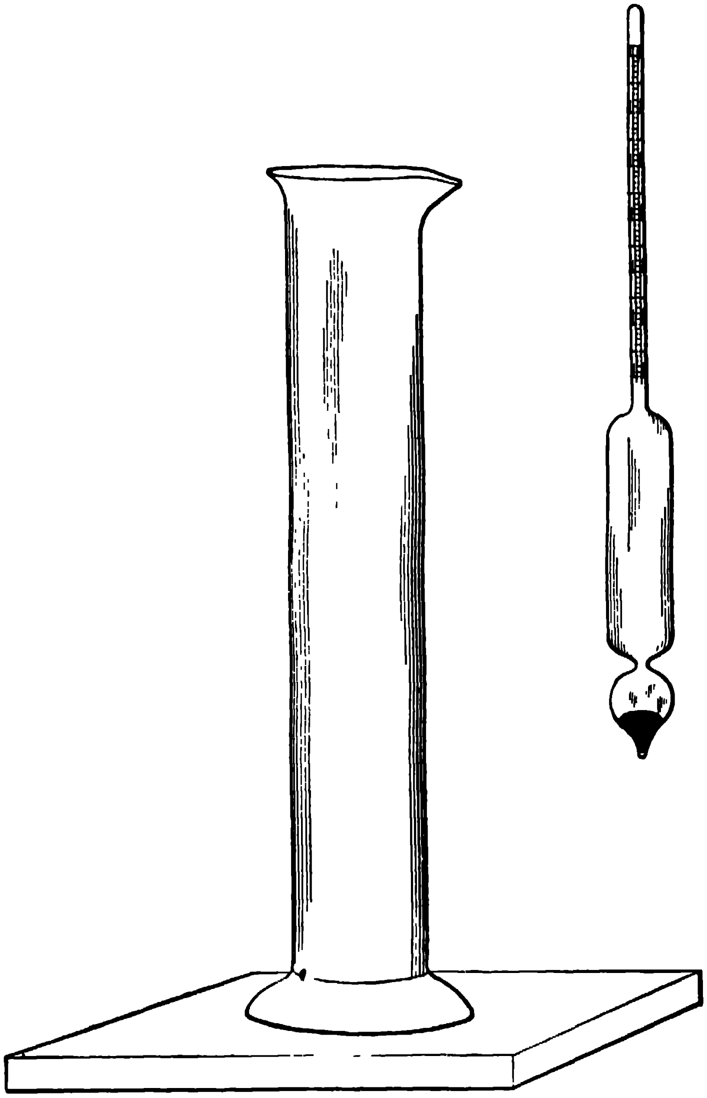

FIG. 225.—Hydrometer Jar and Hydrometer.

With the Twaddell hydrometer, employed largely in England, the density of pure distilled water is represented by 0° and the scale shows 200° between the specific gravity 1. and specific gravity 2. so that the specific gravity of the liquid may be calculated by multiplying the number of degrees registered on the scale by 5 and adding the product to 1.000. Thus the density of a liquid indicating 100° Twaddell (or Tw.) would be 100×5+1.000, or 1.500.

In the following table is given a comparison of the arbitrary scales of Baumé and Twaddell with the corresponding specific gravity:

TABLE XXXVIII.—COMPARISON OF HYDROMETER SCALES WITH BAUMÉ DEGREES AS THE BASIS.

°Baumé.	°Twaddell.	Specific Gravity.	°Baumé.	°Twaddell.	Specific Gravity.
1	1.4	1.007	36	66.4	1.332
2	2.8	1.014	37	69.	1.345
3	4.4	1.022	38	71.4	1.357
4	5.8	1.029	39	74.	1.370
5	7.4	1.037	40	76.6	1.383
6	9.	1.045	41	79.4	1.397
7	10.4	1.052	42	82.	1.410
8	12.	1.060	43	84.8	1.424
9	13.4	1.067	44	87.6	1.438
10	15.	1.075	45	90.6	1.453
11	16.6	1.083	46	93.6	1.468
12	18.2	1.091	47	96.6	1.483
13	20.	1.100	48	99.6	1.498
14	21.6	1.108	49	102.8	1.514
15	23.2	1.116	50	106.	1.530
16	25.	1.125	51	109.2	1.546
17	26.8	1.134	52	112.6	1.563
18	28.4	1.142	53	116.	1.580
19	30.4	1.152	54	119.4	1.597
20	32.4	1.162	55	123.	1.615
21	34.2	1.171	56	126.8	1.634
22	36.	1.180	57	130.4	1.652
23	38.	1.190	58	134.2	1.671
24	40.	1.200	59	138.2	1.691
25	42.	1.210	60	142.2	1.711
26	44.	1.220	61	146.4	1.732
27	46.2	1.231	62	150.6	1.753
28	48.2	1.241	63	154.8	1.774
29	50.4	1.252	64	159.2	1.796
30	52.6	1.263	65	163.8	1.819
31	54.8	1.274	66	168.4	1.842
32	57.	1.285			
33	59.4	1.297			
34	61.6	1.308			
35	64:	3.320			

Examination of Soap.—The chemical analysis of soap in process of manufacture, as a rule, does not extend beyond the determination of free or unsaponified fat and of free or uncombined alkali. The physical tests for these bodies suffice for all practical purposes and can be made by a competent soap-boiler. The complete saponification which may be indicated by physical tests at the soap-kettle is not, however, chemically complete, the amount of unsaponified matter in particular being considerable, especially with rosined soap, while with all soap the presence of free alkali may be indicated by spotting the freshly-cut surface with phenolphthalein. The unsaponified matter of well-made grained soap is largely unsaponifiable matter.

The examination of the finished product comprises the following essential determinations, to which may be added other determinations according to the nature of the soap and the information desired: Moisture, mixed fatty acids, combined alkali. These determinations give the actual soap (anhydrous) and water present in the sample. The determinations of unsaponified fat and uncombined alkali indicate the thoroughness of saponification and serve to distinguish between a cold-process and a grained soap. The determination of soda-ash and sodium silicate is a valuable index in the valuation of laundry soap which is the most common vehicle for these bodies. The separation of the mixed fatty acids into fat and rosin acids, in case of a rosined soap, and the examination of the separated fatty acids, are important to determine the nature and origin of the soap-stock used. With special soaps the following determinations may be mentioned: Glycerin in cold process soap; glycerin and sugar in transparent soap; talc in milled and other grades of soap, and numerous tests for the determination of substance to a high degree erroneously claimed to impart beneficial properties to the soap itself.

Moisture.—The readiness with which highly hydrated soap, as grained, settled soap, dries presents some difficulty in an accurate determination of the amount of water originally present. With a fresh cake the sample may be taken by cutting the cake through the middle into two parts and collecting the shavings cut from the entire freshly-cut surface. If the cake has been exposed for some time the sample should be cut from the interior of the cake. Care should be observed that all portions of the cake weighed out for analysis should be of the same consistency, as for example, the determination of one ingredient should not be made in a portion taken from the exterior, while another determination is made from a portion taken from the interior of the bar or cake. In the determination of the moisture, from 2 to 3 grams of the shavings are weighed into a small tared beaker and placed in the hot closet where it is allowed to dry to a constant weight. The loss in weight is water.

Mixed Fatty Acids.—Weigh out into a beaker of convenient size 5 grams of the sample. Dissolve the soap in a small amount of hot water. It is important to observe the behavior of the soap during dissolution and the character of the resulting solution. Inert mineral matter as talc, silex, etc., collects on the bottom of the beaker; organic matter as starch makes the soap solution turbid. Starch can be detected by the blue coloration produced with iodin solution and its presence may be confirmed by the microscope. If mineral matter is present this may be washed thoroughly with hot water to remove adhering traces of soap and the wash-water added to the soap solution. The mineral matter may then be collected on a tarred filter and dried. Add to the soap solution an excess of semi-normal sulphuric acid, using methyl orange as indicator. Place the beaker in the steam-bath and heat with stirring until the separated fatty acids have collected in an oily layer. Cool the contents of the beaker until the fatty acids have solidified. Remove the liquid by decantation, and if any mineral matter is present separate the same without loss of the fatty acids. The fatty acids may now be remelted with hot water, cooled, and washed to remove adhering traces of mineral acid, the wash-water being added to the decanted liquid. The fatty acids are now collected on a tared filter, dried, cooled, and weighed.

With soap containing sodium silicate, gelatinous silicic acid separates on the addition of sulphuric acid and remains mixed with the fatty acids. The separation of the fatty acids from the gelatinous silicic acid may be effected by placing the funnel containing the filter on which the mixed fatty acids are collected, in a tared beaker and transferring the whole to the hot closet, whereupon as the filter dries the fatty acids melt, pass through and collect in the beaker. The filter-paper and residue are washed thoroughly with hot alcohol or petroleum ether. The solvent is evaporated and the dried residue and fatty acids respectively are weighed.

Total Alkali.—The decanted liquid and wash-water from the separated fatty acids contains the total alkali existing in the soap as carbonate, silicate, hydrate, and in combination with fat anhydrides, as sulphate; it will also contain glycerin, starch if present, the excess of standard sulphuric acid and soluble fatty acids (which may be disregarded) in small amount if cocoanut or palm-kernel oils be a stock ingredient of the soap. The liquid is titrated with semi-normal caustic soda using as indicator the methyl orange already added. From the number of cubic centimeters of $\frac{N}{2}$ H_2SO_4 required for neutralization calculate the percentage of total alkali as Na_2O. The percentage of sodium carbonate, sodium tetrasilicate and sodium hydrate, when determined, is expressed as Na_2O. This total percentage subtracted from the percentage of total alkali gives the percentage of Na_2O combined with fat anhydrides.

Free Alkali.—The determination of free or uncombined alkali requires the extraction of the sample with hot neutral absolute alcohol. For this purpose an extraction apparatus, of which there are numerous forms, may be used, or the extraction may be made in a beaker in the following manner: Weigh out 3 to 5 grams in a beaker and digest on the steam-bath with absolute alcohol with occasional stirring, using a watch-glass to cover the beaker. The hot solution is filtered rapidly through a tared filter to avoid gelatinization of the alcoholic-soap solution on cooling. The extraction is repeated with fresh alcohol to insure thoroughness and the filter must be well washed. Titrate the filtrate with $\frac{N}{10}$ hydrochloric acid using phenolphthalein as indicator. Error by the alcohol method arises from the simultaneous presence of both free alkali and uncombined fat. When high-grade soaps with little or no free fat are examined the method is fairly satisfactory. The determination is a difficult problem in some instances. As the conditions necessary to insure the stability of the soap are the same as those necessary to insure the absolute saponification of the glycerides by the free alkali present, the alcohol method is certainly not to be recommended for cold-process or other soap wherein one has reason to suspect the existence of both free fat and free alkali.

If it is desired to obtain accurate results on the caustic and carbonate separately the preliminary drying of the soap introduces an error, since the caustic alkali will take up carbon dioxide from the air unless the drying is done out of contact with air. It is quite a troublesome process to filter an alcoholic soap solution if one is not provided with appliances to keep the funnel hot during filtration.

Under these conditions it were better to express, first the percentage of total alkali, then the alkali combined with the mixed fatty acids, and if the alkali present as silicate and carbonate be carefully examined some idea may be gained of that present as hydrate.

To use the alcohol method with accuracy it is, therefore, necessary to dry the soap in an atmosphere free from carbon dioxide, extract the free fat with petroleum ether, dissolve the extracted soap in alcohol and titrate as before.

In the following method Devine determines the free alkali and eliminates filtration. For this method it is necessary to provide three standard solutions:

I. Hydrochloric acid, $\frac{N}{10}$ (for standardizing II).

II. Caustic soda, $\frac{N}{10}$, in alcohol.

III. Stearic acid, $\frac{N}{10}$, in alcohol.

II. and III. should be exactly equivalent one to the other, titrated warm with phenolphthalein as indicator.

Two grams of soap (which needs no drying) is weighed into a round-bottomed flask of about 300 cubic centimeters capacity, and 50 cubic centimeters of alcohol poured upon it. $\frac{N}{10}$ stearic acid is now run in from a burette in amount judged to be sufficient to neutralize the free alkali in 2 grams of the soap, some phenolphthalein added, and the flask then stoppered with a cork stopper, through which pass a glass-tube about 30 inches long and of about ¼ inch internal diameter, the lower end ground to a point on a grindstone, and the purpose of which is to serve as a reflux condenser. The flask and contents are placed on a steam-bath and heated 30 minutes, at the expiration of which time the solution should be quite clear and show no alkali with the phenolphthalein. If the solution turns red during the boiling, showing that an insufficient quantity of stearic acid has been added at first, add more of that solution until the color disappears, then several cubic centimeters in excess, and heat 20 minutes further. The flask is now removed from the bath and after a few minutes' cooling is titrated with $\frac{N}{10}$ caustic soda. The difference between the number of cubic centimeters of stearic-acid solution added and the number of cubic centimeters of caustic soda used to back titrate is equivalent to the total free alkali present.

While the first flask is heating weigh out in a similar flask 2 grams of soap and add 50 cubic centimeters of alcohol and place on the steam-bath. When the first test is finished, calculate roughly the total alkali, assuming the total quantity to be carbonate. Now add to the second flask an amount of 10 per cent barium chloride solution sufficient to precipitate the alkali, heat a few minutes, add phenolphthalein, and titrate with $\frac{N}{10}$ stearic acid. The titration must take place slowly and with thorough agitation of the liquid for the reason that the sodium or potassium hydroxide reacts with the barium chloride added and forms sodium chloride and barium hydroxide. The latter is not very soluble in the alcoholic liquid and sufficient time and pains must be taken to insure its complete neutralization by the stearic acid. A blank test should be made on 50 cubic centimeters of the alcohol, since this frequently contains carbon dioxide, and the number of tenths of cubic centimeters of $\frac{N}{10}$ caustic soda necessary to neutralize the free acid in this quantity of alcohol added to the reading of the stearic-acid burette in the second test. This corrected reading gives the number of cubic centimeters of $\frac{N}{10}$ stearic acid used to neutralize the caustic alkali in 2 grams of soap. The difference between the total alkali found and the caustic will, of course, give the carbonate.

Alkaline Residue Insoluble in Alcohol.—The residue insoluble in alcohol remaining on the filter or in the extraction-tube is then extracted with hot water and the extract titrated with semi-normal sulphuric acid using methyl orange as indicator. After obtaining the per cent of alkaline residue insoluble in alcohol, which comprises both silicate and carbonate, and borate if present, the anhydrous silica may be obtained separately by digestion with hydrochloric acid as follows:

Silica.—50 grams of soap are ignited in a platinum dish until all the carbonaceous matter is burned off. The residual ash is treated with concentrated hydrochloric acid, and the whole evaporated to complete dryness over a water-bath. This decomposes the silicates into silica (SiO_2) and chlorides of the bases. The latter, which are soluble, are dissolved with warm dilute hydrochloric acid and washed on to a filter. The residue is washed with warm water, dried on the filter at 100° C., and weighed as silica, which may be calculated into sodium silicate.

To insure the absence of talc or ground quartz from the silicious residue it is necessary to make a separate test for insoluble matter. If insoluble mineral matter is found to be present its separation may be effected as follows: Dissolve a weighed portion of the soap in water and allow the insoluble matter to subside. Decant the solution and wash the residue thoroughly. The soap solution may be evaporated to dryness and ignited at once, or the fatty acids may be separated, preferably by means of hydrochloric acid, and the filtrate evaporated to dryness and digested as described with concentrated hydrochloric acid for the dehydration of the silicic acid.

Much uncertainty in the transformation of silica into sodium silicate and into sodium-silicate solution arises from ignorance of the proportions of sand and soda-ash used in its manufacture, and of the proportions of sodium oxide and silica in the compound contained in the resulting solution which, as has been previously stated, as used in soap-manufacture is of a density of 40° Bé. As an excess of soda-ash is used in the manufacture of sodium silicate the commercial product contains a greater or less amount of uncombined carbonate of soda. The sodium silicate applicable for soap-manufacture is considered to be a tetrasilicate, $Na_2O_4SiO_2$, in which the ratio of soda (Na_2O) and silicic acid (SiO_2) is as follows: Na_2O, 20.53 per cent; SiO_2, 79.47 per cent. From analyses of sodium-silicate solutions of known density and calculations therefrom, a writer in *Der Seifenfabricant* has compiled the following table based upon the composition of the alkaline silicate as a tetrasilicate, which may be found useful for the reduction of the percentage of anhydrous silicic acid found by analysis to its corresponding tetrasilicate and to the weight of the original sodium silicate solution containing it.

TABLE XXXIX.—SODIUM (TETRA) SILICATE IN SODIUM-SILICATE SOLUTIONS.

Degrees Bé.	Specific Gravity.	Per Cent SiO_2.	Per Cent Na_2O.	Per Cent $Na_2O_4SiO_2$.	Degrees Bé.	Specific Gravity.	Per Cent SiO_2.	Per Cent Na_2O.	Per Cent $Na_2O_4SiO_2$.
0	1.0000	...	...	...	21	1.1667	14.3	3.8	18.1
1	1.0070	0.7	0.2	0.9	22	1.1764	15.1	4.0	19.1
2	1.0139	1.3	0.4	1.7	23	1.1856	15.8	4.1	19.9
3	1.0209	2.0	0.6	2.6	24	1.1956	16.5	4.3	20.8
4	1.0278	2.6	0.8	3.4	25	1.2051	17.3	4.5	21.8
5	1.0352	3.3	0.9	4.2	26	1.2153	18.0	4.7	22.7
6	1.0426	4.0	1.1	5.1	27	1.2251	18.7	4.9	23.6
7	1.0498	4.7	1.3	6.0	28	1.2356	19.4	5.0	24.4
8	1.0579	5.5	1.5	7.0	29	1.2461	20.1	5.2	25.3
9	1.0657	6.2	1.7	7.9	30	1.2569	20.8	5.3	26.1
10	1.0731	6.9	1.9	8.8	31	1.2677	21.5	5.5	27.0
11	1.0811	7.7	2.0	9.7	32	1.2787	22.2	5.7	27.9
12	1.0891	8.5	2.2	10.7	33	1.2905	23.0	5.8	28.8
13	1.0969	9.1	2.4	11.5	34	1.3012	23.7	6.0	29.7
14	1.1051	9.7	2.5	12.2	35	1.3132	24.4	6.2	30.6
15	1.1135	10.3	2.7	13.0	36	1.3248	25.0	6.3	31.3
16	1.1220	10.9	2.8	13.7	37	1.3371	25.7	6.4	32.1
17	1.1311	11.5	3.0	14.5	38	1.3490	26.3	6.6	32.9
18	1.1398	12.2	3.2	15.4	39	1.3616	27.0	6.8	33.8
19	1.1487	12.9	3.4	16.3	40	1.3751	27.7	7.0	34.7
20	1.1576	13.6	3.6	17.2					

The total residue insoluble in alcohol may contain sodium carbonate, sodium silicate, sodium biborate, and insoluble matter as talc. Talc is determined separately as insoluble matter. Sodium biborate may be present, but the high cost militates against its use even in so-called borax soaps. A qualitative test for borax may be made as follows: If the solution to be tested has to be evaporated it first must be made alkaline, as boric acid is volatile with steam. Add to the solid substance concentrated H_2SO_4 and alcohol. Warm the mixture, set fire to it, and note whether the edges of the flame are colored green. The color is best seen at the moment the alcohol is lighted. In the presence of chlorides the test is unreliable, as ethyl chloride, which also tinges the flame green, is formed by their action on alcohol. Another test is to acidify the solution slightly with HCl and partly dip a piece of turmeric-paper in it and dry. Note whether the dipped part assumes a red tint. If the HCl is not very dilute, it imparts a brownish-black color to the turmeric-paper which obscures the boric-acid test.

The alkaline residue, as a rule, consists of the silicate and carbonate of soda, both of which are determined volumetrically and expressed as Na_2O. The silica from sodium silicate having been determined, the Na_2O required to form the tetrasilicate is subtracted from the total Na_2O, leaving the remainder to be transformed into sodium carbonate. The water associated with the tetrasilicate is included in percentage of moisture.

Insoluble Matter.—The insoluble matter present in a soap refers to that portion which is insoluble in water, and usually consists of silica (ground quartz, sand, or silex) or talc. The character and relative amount of insoluble matter may be observed on dissolving the portion of the sample intended for the determination of mixed fatty acids and combined alkali in water, and when convenient its determination can be made in this portion of the sample. Its determination is important in the examination of sand or scouring-soap and soap-powder. A suitable weight of the sample is dissolved in water in a beaker of convenient size. After complete solution of the soap and soluble mineral matter as soda-ash and sodium silicate and deposition of the insoluble portion, the liquid is decanted off and the insoluble matter is washed thoroughly, preferably in the beaker, and is finally washed upon a tared filter, after which it is dried and weighed.

The insoluble matter includes also unsaponifiable mineral or hydrocarbon oil obtained from petroleum. To determine this a weighed portion of the soap is dissolved in water and the solution transferred to a separatory funnel. To this ether is added in the cold and the contents of the funnel are well shaken. Allow to stand until the ethereal solution separates, when it is run off into a tared beaker from which the solvent is evaporated on the steam-bath. Mineral oil is added to soap either to increase the cleansing action of the soap by the solvent action of the mineral oil upon fatty matter, or is added as a filler or as a binding agent as mineral soap-stock.

Rosin.—The ethyl ethers of fatty acids are most readily formed by acting on an alcoholic solution of the latter with hydrochloric-acid gas, the HCl merely serving to remove the water formed by the combination. This reaction Twitchell found to be practically complete where the alcohol employed is absolute and the HCl gas is passed in to saturation.

On attempting to etherify resin acids (common rosin) in the same way, Twitchell found that no combination takes place between the alcohol and acid, and that when the solution is kept cool the resin acid is entirely unacted upon and can again be separated by diluting with water and boiling to collect the precipitate.

The determination of rosin by the Twitchell method, which is the one generally used, depends on the fact that by the means indicated all the fatty acids are combined to form ethers which are neutral in alcoholic solution and unacted upon by alkalis in the cold, while the rosin remains unchanged, reacts acid in alcoholic solution with phenolphthalein, and combines readily with caustic alkali to form a soluble soap. It is, therefore, simply necessary to effect combination of the fatty acids with alcohol, while the resin acids may be titrated with standard caustic-soda solution, using phenolphthalein as indicator, or they may be combined with caustic alkali and the rosin soap thus formed separated from the unsaponified fatty ethers by extraction with naphtha in a separatory funnel.

The gravimetric method is carried out as follows: 2 or 3 grams of the mixed fat and resin acids obtained as previously described are dissolved in ten times their volume of absolute alcohol in a flask and dry HCl gas passed through in a moderate stream. The flask is set in a vessel with water to keep it cool. The HCl is rapidly absorbed, and after about 45 minutes the ethers separate, floating on the solution, and no more HCl is absorbed. The flask is removed and allowed to stand a half hour longer to ensure a complete combination of the alcohol and fatty acid. It is then diluted with about five times its volume of water and boiled until the acid solution is clear, the ethers with rosin in solution floating on the top. To this is added some naphtha and the whole transferred to a separatory funnel, the flask being washed out with naphtha. The acid solution is then run off and the naphtha solution, which should measure about 50 cubic centimeters, is washed once with water and then treated in the funnel with a solution of 5 grams KOH and 5 cubic centimeters of alcohol in 50 cubic centimeters of water and agitated. The rosin is immediately saponified and the two layers separate completely. The solution of rosin soap can then be run off, treated with acid, the rosin collected in any manner desired, dried and weighed. A second washing of the soap with naphtha is hardly necessary, as very little remains after the first extraction.

The first stages of the volumetric method are similar to those of the gravimetric, with the exception that the contents of the flask are washed into the separatory funnel with ether instead of naphtha, and the ether solution in the funnel is then thoroughly washed with water until the wash-water is no longer acid; 50 cubic centimeters of alcohol previously neutralized are then added and the solution titrated with standard NaOH solution, using phenolphthalein as indicator.

The determination of rosin as devised by Twitchell gives the most satisfactory results, which, while not absolutely accurate, are within the limits ordinarily required in soap analysis. The wide variation in the combining weight of rosin imparts uncertainty to the results obtained by the volumetric method, while by the gravimetric method low results arise from failure to extract unsaponifiable portions of the rosin from the petroleum-ether layer. Lewkowitsch believes that under the action of hydrochloric acid the resin acids appear to undergo some destruction with the formation of acids of lower molecular weight, which would account to a degree for the low results by the volumetric method; while in the gravimetric method, low results arise not only from the reason stated, but from the passage of these secondary products into the aqueous solution without being extracted by the petroleum ether.

As a qualitative test for rosin Gottlieb's method is reliable and easily made.

The soap is dissolved in water and heated to boiling. A strong solution of $MgSO_4$ is added until the fatty acids are completely precipitated. The magnesium resinates remain in solution. After boiling 2 to 3 minutes the solution is filtered and the hot filtrate acidified with H_2SO_4. In the presence of rosin the liquid becomes turbid, due to the separated resin acids. The boiling should be continued for half an hour, to make sure that the turbidity is due to the resin acids and not to volatile fatty acids.

Barfoed recommends as a qualitative test for rosin to dissolve the soap or mixed fat and resin acids in 80 per cent alcohol, and add an excess of an alcoholic solution of $CaCl_2$ with ammonia to alkaline reaction. After cooling, the precipitate is washed and filtered. The filtrate contains the calcium salt of the resin acids, together with a slight amount of calcium oleate. If it is now acidified with HCl the resin separates as a soft, gummy, and viscous mass.

Fatty Acids.—The chemical examination of the fatty acids obtained by decomposing the soap with mineral acid, to determine the nature of the stock used, is a difficult problem. Frequently the efforts made to solve it by analysis are not always warranted by the facts. Practical experience in soap-boiling, combined with a knowledge of the chemical and physical characteristics of the soap-stock available or suitable for the manufacture of any given soap and their prevailing market price, the percentage of rosin in the case of a rosined soap and other data obtained in the course of the analysis of the sample, and most important, the physical appearance, purpose, and selling price of the soap, are adequate to indicate the nature of the ingredients used and their probable relative proportions. Facts obtained by analysis combined with the facts enumerated are in every case superior in definiteness and reliability to the results of any chemical or physical examination of the liberated fatty acids, and for all practical and necessary purposes in soap-manufacture are the only facts to be depended upon.

The examination of the fatty acids comprise the observation of their color, odor, and taste, together with the melting and solidifying-points, the alkali absorption or combining weight and the iodin number. A disturbing element in any examination is the fact that fatty acids from a mixture of fats will give chemical and physical constants that approximate those of a single fat. Sole dependence upon chemical analysis in such a case cannot result in other than erroneous conclusions. Bleaching, refining, and compounding, to which much soap-stock is subjected, modify to a greater or less extent the characteristics of the original material. With, however, a mixture of two fats in their natural state, without having undergone any material change by bleaching, refining, etc., it is possible to ascertain with considerable accuracy the nature of the fatty acids by means of the iodin number.

DETERMINATION OF IODIN ABSORPTION BY HÜBL'S METHOD.

Preparation of Reagents.—*Iodin Solution.*—Dissolve 25 grams of pure iodin in 500 cubic centimeters of 95 per cent alcohol. Dissolve 30 grams of mercuric chlorid in 500 cubic centimeters of 95 per cent alcohol. The latter solution, if necessary, is filtered, and then the two solutions mixed. The mixed solution should be allowed to stand 12 hours before using.

Decinormal Sodium Thiosulphate Solution.—Dissolve 24.8 grams of chemically pure sodium thiosulphate freshly pulverized as finely as possible and dried between filter- or blotting-paper and dilute with water to 1 litre at the temperature at which the titrations are to be made.

Starch Paste.—One gram of starch is boiled in 200 cubic centimeters of distilled water for 10 minutes and cooled to room temperature.

Solution of Potassium Iodid.—One hundred and fifty grams of potassium iodid are dissolved in water and made up to 1 litre.

Solution of Potassium Dichromate.—Dissolve 3.874 grams of chemically pure potassium dichromate in distilled water and make the volume up to 1 litre at the temperature at which the titrations are to be made and standardized against pure iron wire.

Standardizing the Sodium-thiosulphate Solution.—Place 20 cubic centimeters of the potassium-dichromate solution, to which has been added 30 cubic centimeters of the solution of potassium iodid, in a glass-stoppered flask. Add to this 5 cubic centimeters of strong hydrochloric acid. Allow the solution of sodium-thiosulphate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste, and with constant shaking continue to add the sodium-thiosulphate solution until the blue color just disappears. The number of cubic centimeters of thiosulphate solution used multiplied by 5 is equivalent to 1 gram of iodin.

Example.—Twenty cubic centimeters $K_2Cr_2O_7$ solution required 16.2 cubic centimeters sodium thiosulphate; then $16.2 \times 5 = 81$ = number of cubic centimeters of thiosulphate solution, equivalent to 1 gram of iodin. Then 1 cubic centimeter thiosulphate solution =0.0124 gram of iodin. Theory for decinormal solution of sodium thiosulphate, 1 cubic centimeter =0.0127 gram of iodin.

Weighing the Sample.—Weigh about 1 gram of fat or 0.500 gram of oil* or a corresponding amount of the fatty acids on a small watch-crystal or any light weighing-glass. The fat is first melted, mixed thoroughly, poured onto the crystal, and allowed to cool.

Introduce the watch-crystal into a wide-mouth 16-ounce bottle with ground-glass stopper.

Absorption of Iodin.—The fat or oil in the bottle is dissolved in 10 cubic centimeters of chloroform. After complete solution has taken place, 30 cubic centimeters of the iodin solution are added in the case of fats, or from 40 to 50 cubic centimeters † in the case of oils. Place the bottle in a dark place and allow to stand, with occasional shaking, for 3 hours.‡ There must be a large excess of iodin or the results will not be satisfactory.

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* With drying-oils which have a very high absorbent power 0.100 to 0.200 gram should be taken.

† F. Ulzer, Jour. Soc. Chem. Ind., 1898, **17**, 276, says iodin should be in excess about twice the amount that is absorbed. The solution loses strength with age, but can be used so long as 35 cubic centimeters of decinormal thiosulphate neutralize 25 cubic centimeters iodin solution. R. Henriques, Zeitsch. anal. Chem., 1901, **40**, 429, says iodin should be used in excess at least 60 per cent of amount added.

‡ The time allowed does not give the complete iodin absorption power of an oil or

Titration of the Unabsorbed Iodin.—Add 20 cubic centimeters of the potassium-iodid solution and then 100 cubic centimeters of distilled water to the contents of the bottle. Wash any iodin which may be noticed upon the stopper of the bottle back into the bottle with the potassium-iodid solution. Titrate the excess of iodin with the sodium-thiosulphate solution, which is run in gradually, with constant shaking, until the yellow color of the solution has almost disappeared. Add a few drops of starch paste, and continue the titration until the blue color has entirely disappeared. Toward the end of the reaction stopper the bottle and shake violently, so that any iodin remaining in solution in the chloroform may be taken up by the potassium-iodid solution. The excess of sodium-thiosulphate solution should be sufficient to prevent a reappearance of any blue color in the flask for 5 minutes.

Setting the Value of Iodin Solution by Thiosulphate Solution.—At the time of adding the iodin solution to the fat, two bottles of the same size as those used for the determination should be employed for conducting the operation described above, but without the presence of any fat. In every other respect the performance of the blank experiments should be just as described. These blank experiments must be made each time the iodin solution is used.

Example Blank Determinations.—Thirty cubic centimeters iodin solution required 46.55 cubic centimeters of sodium-thiosulphate solution. Thirty cubic centimeters iodin solution required 46.65 cubic centimeters of sodium-thiosulphate solution. Mean, 46.6 cubic centimeters.

Per cent of iodin absorbed:

Weight of fat taken........................	1.0479 grams		
Quantity of iodin solution used............	40.0	cubic	centimeters
Thiosulphate equivalent to iodin used......	62.1	"	"
Thiosulphate equivalent to remaining iodin..	30.2	"	"
Thiosulphate equivalent to iodin absorbed..	31.9	"	"

Per cent of iodin absorbed, $31.9 \times 0.0124 \times 100 \div 1.0479 = 37.75$.

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fat and cannot be compared with determinations where six to twelve hours have been used. It gives very satisfactory comparative results, but the time factor must be very closely adhered to.

Glycerin in Soap.—The presence of glycerin in considerable amount in a soap that has not been milled is indisputable evidence that it has not been made by the grained process wherein waste lye is withdrawn, but is according to its texture and other characteristics either a cold-process or semi-boiled soap. The preparation of the solution in which glycerin is determined involves the decomposition of a weighed quantity of the soap with mineral acid, preferably sulphuric, and the removal of all organic matter that would be oxidized by the bichromate solution. The filtrate containing the glycerin may be concentrated to a convenient bulk, taking care to avoid loss of glycerin, or the determination may be made directly. The determination of glycerin in transparent soap is a difficult problem owing to the presence in greater of less amount of sugar. Freyer determines the sugar as follows: 16–28 grams of the soap are dissolved in 50–100 cubic centimeters of water and 10 per cent barium-chloride solution added, with vigorous shaking, till the formation of foam ceases. The liquid is then made up to 260 cubic centimeters, 10 cubic centimeters being allowed for the volume of the barium precipitate, and the filtrate is polarized, then examined for reducing-sugar, inverted, and again polarized, or the sugar, after inversion, determined gravimetrically.

Donath and Mayrhofer determine the glycerin by adding to the filtrate from the fatty acids a quantity of slaked lime sufficient to combine with the sugar present and an equal quantity of washed and ignited sand. The solution is concentrated to the consistency of a syrup. The residue is extracted with equal volumes of alcohol and ether. The extract will contain all the glycerin free from sugar and may be determined by either the acetin or bichromate method. Qualitative test for sugar may be made with Fehling's solution.

Chlorides and Sulphates.—For the determination of chlorides a weighed portion of the soap is decomposed with nitric acid and the filtrate from the fatty acids made up to a convenient volume. An aliquot portion is taken for titration. After neutralizing with sodium carbonate, titrate with $\frac{N}{10}$ $AgNO_3$ solution, using potassium chromate as indicator as before described.

To determine sulphates, the soap is decomposed with hydrochloric acid and to the filtrate from the fatty acids, while hot, an excess of $BaCl_2$ solution is added. The precipitate is dried on the filter to constant weight, the tare of the filter deducted, and the residue calculated as sodium sulphate.[1]

Statement of Results.—The following outline represents a form for the statement of the results of the analysis

Ingredients.	Per Cent.	Per Cent.	Per Cent.
Moisture	.....	.....	25.08
Free fat, rosin, mineral oil, etc.	0.97		
Free alkali, NaOH	0.10		
Sodium carbonate, Na_2CO_3	1.82		
Sodium silicate, $Na_2O_4SiO_2$	1.40		
Total unsaponified matter	.....	.....	4.29
Fat anhydrides	37.94		
Rosin	26.30		
Total anhydrides	.....	64.24	
Combined alkali, Na_2O	.....	6.65	
Anhydrous soap	.....	.....	70.89
Total	.....	.....	100.26

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[1] Fat exists in soap as fat anhydrides in combination with sodium oxide, but is separated and weighed as fatty acids. To transform fatty acids into fat anhydrides multiply the former by the factor 0.97; to transform fat anhydrides into glycerides divide the former by 0.9.

APPENDIX.

Table XL. Comparison of Centigrade Degrees with Fahrenheit Degrees as a Basis.
Table XLI. Comparison of Fahrenheit Degrees with Centigrade Degrees as a Basis.
Table XLII. Specific Gravity of Sulphuric Acid.
Table XLIII. Specific Gravity of Hydrochloric Acid.
Table XLIV. Number of Gallons in Cisterns and Tanks.
Table XLV. Value of Commercial Products as Compared with Chemically Pure Products.
Table XLVI. Equivalent Prices for Soda-ash.
Table XLVII. Equivalent Prices for Caustic Soda.
Table XLVIII. Pounds of Tallow Saponified by 100 Pounds of Caustic Soda Lye of Different Densities Made of the Grades of Caustic Indicated (Lamborn).
Table XLIX. Specific Gravity of Aqueous Solutions of Glycerin.
Table L. Specific Gravity of Aqueous Solutions of Glycerin (Skalweit).
Table LI. Pressure and Temperature of Saturated Steam.

TABLE XL.—COMPARISON OF CENTIGRADE DEGREES WITH FAHRENHEIT DEGREES AS A BASIS.

$$^\circ\text{Fahr.} = {}^\circ\text{C.} \times \tfrac{9}{5} + 32.$$

F.	C.	F.	C.	F.	C.	F.	C.	F.	C.
1	17.2	44	6.7	87	30.6	129	53.9	171	77.2
2	16.7	45	7.2	88	31.1	130	54.4	172	77.8
3	16.1	46	7.8	89	31.7	131	55.0	173	78.3
4	15.6	47	8.3	90	32.2	132	55.6	174	78.9
5	15.0	48	8.9	91	32.8	133	56.1	175	79.4
6	14.4	49	9.4	92	33.3	134	56.7	176	80.0
7	13.9	50	10.0	93	33.9	135	57.2	177	80.6
8	13.3	51	10.6	94	34.4	136	57.8	178	81.1
9	12.8	52	11.1	95	35.0	137	58.3	179	81.7
10	12.2	53	11.7	96	35.6	138	58.9	180	82.2
11	11.7	54	12.2	97	36.1	139	59.4	181	82.8
12	11.1	55	12.8	98	36.7	140	60.0	182	83.3
13	10.6	56	13.3	99	37.2	141	60.6	183	83.9
14	10.0	57	13.9	100	37.8	142	61.1	184	84.4
15	9.4	58	14.4	101	38.3	143	61.7	185	85.0
16	8.9	59	15.0	102	38.9	144	62.2	186	85.6
17	8.3	60	15.6	103	39.4	145	62.8	187	86.1
18	7.8	61	16.1	104	40.0	146	63.3	188	86.7
19	7.2	62	16.7	105	40.6	147	63.9	189	87.2
20	6.7	63	17.2	106	41.1	148	64.4	190	87.8
21	6.1	64	17.8	107	41.7	149	65.0	191	88.3
22	5.6	65	18.3	108	42.2	150	65.6	192	88.9
23	5.0	66	18.9	109	42.8	151	66.1	193	89.4
24	4.4	67	19.4	110	43.3	152	66.7	194	90.0
25	3.9	68	20.0	111	43.9	153	67.2	195	90.6
26	3.3	69	20.6	112	44.4	154	67.8	196	91.1
27	2.8	70	21.1	113	45.0	155	68.3	197	91.7
28	2.2	71	21.7	114	45.6	156	68.9	198	92.2
29	1.7	72	22.2	115	46.1	157	69.4	199	92.8
30	1.1	73	22.8	116	46.7	158	70.0	200	93.3
31	0.6	74	23.3	117	47.2	159	70.6	201	93.9
32	0.0	75	23.9	118	47.8	160	71.1	202	94.4
33	0.6	76	24.4	119	48.3	161	71.7	203	95.0
34	1.1	77	25.0	120	48.9	162	72.2	204	95.6
35	1.7	78	25.6	121	49.4	163	72.8	205	96.1
36	2.2	79	26.1	122	50.0	164	73.3	206	96.7
37	2.8	80	26.7	123	50.6	165	73.9	207	97.2
38	3.3	81	27.2	124	51.1	166	74.4	208	97.8
39	3.9	82	27.8	125	51.7	167	75.0	209	98.3
40	4.4	83	28.3	126	52.2	168	75.6	210	98.9
41	5.0	84	28.9	127	52.8	169	76.1	211	99.4
42	5.6	85	29.4	128	53.3	170	76.7	212	100.0
43	6.1	86	30.0						

TABLE XLI.—COMPARISON OF FAHRENHEIT DEGREES WITH CENTIGRADE DEGREES AS A BASIS.

$$°\text{C.} = (°\text{Fahr.} - 32)\tfrac{5}{9}$$

FOR TEMPERATURES BELOW THE FREEZING-POINT OF WATER.

C.	F.	C.	F.	C.	F.	C.	F.	C.	F.
—		-		-	—	—		—	—
°	°	°	°	°	°	°	°	°	°
40	40.0	31	23.8	22	7.6	16	3.2	7	19.4
39	38.2	30	22.0	21	5.8	15	5.0	6	21.2
38	36.4	29	20.2	20	4.0	14	6.8	5	23.0
37	34.6	28	18.4	19	2.2	13	8.6	4	24.8
36	32.8	27	16.6	18	0.4	12	10.4	3	26.6
35	31.0	26	14.8	17.778	0.0	11	12.2	2	28.4
34	29.2	25	13.0	--	+	10	14.0	1	30.2
33	27.4	24	11.2		°	9	15.8	0	32.0
32	25.6	23	9.4	17	1.4	8	17.6		

FOR TEMPERATURES ABOVE THE FREEZING-POINT OF WATER.

C.	F.	C.	F.	C.	F.	C.	F.	C.	F.
+	+	+	+	+	+	+	+	+	+
°	°	°	°	°	°	°	°	°	°
1	33.8	26	78.8	51	123.8	76	168.8	101	213.8
2	35.6	27	80.6	52	125.6	77	170.6	102	215.6
3	37.4	28	82.4	53	127.4	78	172.4	103	217.4
4	39.2	29	84.2	54	129.2	79	174.2	104	219.2
5	41.0	30	86.0	55	131.0	80	176.0	105	221.0
6	42.8	31	87.8	56	132.8	81	177.8	106	222.8
7	44.6	32	89.6	57	134.6	82	179.6	107	224.6
8	46.4	33	91.4	58	136.4	83	181.4	108	226.4
9	48.2	34	93.2	59	138.2	84	183.2	109	228.2
10	50.0	35	95.0	60	140.0	85	185.0	110	230.0
11	51.8	36	96.8	61	141.8	86	186.8	111	231.8
12	53.6	37	98.6	62	143.6	87	188.6	112	233.6
13	55.4	38	100.4	63	145.4	88	190.4	113	235.4
14	57.2	39	102.2	64	147.2	89	192.2	114	237.2
15	59.0	40	104.0	65	149.0	90	194.0	115	239.0
16	60.8	41	105.8	66	150.8	91	195.8	116	240.8
17	62.6	42	107.6	67	152.6	92	197.6	117	242.6
18	64.4	43	109.4	68	154.4	93	199.4	118	244.4
19	66.2	44	111.2	69	156.2	94	201.2	119	246.2
20	68.0	45	113.0	70	158.0	95	203.0	120	248.0
21	69.8	46	114.8	71	159.8	96	204.8	121	249.8
22	71.6	47	116.6	72	161.6	97	206.6	122	251.6
23	73.4	48	118.4	73	163.4	98	208.4	123	253.4
24	75.2	49	120.2	74	165.2	99	210.2	124	255.2
25	77.0	50	122.0	75	167.0	100	212.0	125	257.0

For Temperatures Above the Freezing-point of Water—*Continued.*

C.	F.	C.	F.	C.	F.	C.	F.	C.	F.
+°	+°	+°	+°	+°	+°	+°	+°	+°	+°
126	258.8	171	339.8	216	420.8	261	501.8	306	582.8
127	260.6	172	341.6	217	422.6	262	503.6	307	584.6
128	262.4	173	343.4	218	424.4	263	505.4	308	586.4
129	264.2	174	345.2	219	426.2	264	507.2	309	588.2
130	266.0	175	347.0	220	428.0	265	509.0	310	590.0
131	267.8	176	348.8	221	429.8	266	510.8	311	591.8
132	269.6	177	350.6	222	431.6	267	512.6	312	593.6
133	271.4	178	352.4	223	433.4	268	514.4	313	595.4
134	273.2	179	354.2	224	435.2	269	516.2	314	597.2
135	275.0	180	356.0	225	437.0	270	518.0	315	599.0
136	276.8	181	357.8	226	438.8	271	519.8	316	600.8
137	278.6	182	359.6	227	440.6	272	521.6	317	602.6
138	280.4	183	361.4	228	442.4	273	523.4	318	604.4
139	282.2	184	363.2	229	444.2	274	225.2	319	606.2
140	284.0	185	365.0	230	446.0	275	527.0	320	608.0
141	285.8	186	366.8	231	447.8	276	528.8	321	609.8
142	287.6	187	368.6	232	449.6	277	530.6	322	611.6
143	289.4	188	370.4	233	451.4	278	532.4	323	613.4
144	291.2	189	372.2	234	453.2	279	534.2	324	615.2
145	293.0	190	374.0	235	455.0	280	536.0	325	617.0
146	294.8	191	375.8	236	456.8	281	537.8	326	618.8
147	296.6	192	377.6	237	458.6	282	539.6	327	620.6
148	298.4	193	379.4	238	460.4	283	541.4	328	622.4
149	300.2	194	381.2	239	462.2	284	543.2	329	624.2
150	302.0	195	383.0	240	464.0	285	545.0	330	626.0
151	303.8	196	384.8	241	465.8	286	546.8	331	627.8
152	305.6	197	386.6	242	467.6	287	548.6	332	629.6
153	307.4	198	388.4	243	469.4	288	550.4	333	631.4
154	309.2	199	390.2	244	471.2	289	552.2	334	633.2
155	311.0	200	392.0	245	473.0	290	554.0	335	635.0
156	312.8	201	393.8	246	474.8	291	555.8	336	636.8
157	314.6	202	395.6	247	476.6	292	557.6	337	638.6
158	316.4	203	397.4	248	478.4	293	559.4	338	640.4
159	318.2	204	399.2	249	480.2	294	561.2	339	642.2
160	320.0	205	401.0	250	482.0	295	563.0	340	644.0
161	321.8	206	402.8	251	483.8	296	564.8	341	645.8
162	323.6	207	404.6	252	485.6	297	566.6	342	647.6
163	325.4	208	406.4	253	487.4	298	568.4	343	649.4
164	327.2	209	408.2	254	489.2	299	570.2	344	651.2
165	329.0	210	410.0	255	491.0	300	752.0	345	653.0
166	330.8	211	411.8	256	492.8	301	573.8	346	654.8
167	332.6	212	413.6	257	494.6	302	575.6	347	656.6
168	334.4	213	415.4	258	496.4	303	577.4	348	658.4
169	336.2	214	417.2	259	498.2	304	579.2	349	660.2
170	338.0	215	419.0	260	500.0	305	581.0	350	662.0

FOR TEMPERATURES ABOVE THE FREEZING-POINT OF WATER—*Continued.*

C.	F.	C.	F.	C.	F.	C.	F.	C.	F.
+ °	+ °	+ °	+ °	+ °	+ °	+ °	+ °	+ °	+ °
351	663.8	361	681.8	371	699.8	381	717.8	391	735.8
352	665.6	362	683.6	372	701.6	382	719.6	392	737.6
353	667.4	363	685.4	373	703.4	383	721.4	393	739.4
354	669.2	364	687.2	374	705.2	384	723.2	394	741.2
355	671.0	365	689.0	375	707.0	385	725.0	395	743.0
356	672.8	366	690.8	376	708.8	386	726.8	396	744.8
357	674.6	367	692.6	377	710.6	387	728.6	397	746.6
358	676.4	368	694.4	378	712.4	388	730.4	398	748.4
359	678.2	369	696.2	379	714.2	389	732.2	399	750.2
360	680.0	370	698.0	380	716.0	390	734.0	400	752.0
								450	842.0
								500	932.0

TABLE XLII.—SPECIFIC GRAVITY OF SULPHURIC ACID.

Degrees Baume.	Specific Gravity.	Weight of 1 Cubic Foot in Pounds.	Per Cent O. V.	Pounds O. V. in 1 Cubic Foot.	Degrees Tw.	Per Cent H_2SO_4.
0	1.000	62.37	0.00	0.00	0	0.000
1	1.007	62.81	1.00	0.63	1½	0.935
2	1.014	63.24	2.50	1.58	3	2.337
3	1.021	63.68	3.66	2.33	4	3.422
4	1.028	64.12	5.00	3.21	5½	4.675
5	1.036	64.62	6.00	3.88	7	5.610
6	1.043	65.05	7.00	4.55	8½	6.545
7	1.051	65.55	8.00	5.24	10	7.480
8	1.058	65.99	9.00	5.94	11½	8.415
9	1.066	66.49	10.25	6.82	13	9.584
10	1.074	66.99	11.50	7.70	15	10.752
11	1.082	67.48	12.50	8.44	16½	11.687
12	1.090	67.98	13.60	9.25	18	12.716
13	1.098	68.48	14.70	10.07	19½	13.741
14	1.107	69.04	16.00	11.05	21½	14.960
15	1.115	69.54	17.00	11.82	23	15.895
16	1.124	70.10	18.25	12.79	25	17.064
17	1.133	70.67	19.60	13.85	26½	18.326
18	1.142	71.23	21.00	14.96	28½	19.635
19	1.151	71.79	22.00	15.79	30	20.570
20	1.160	72.35	23.25	16.82	32	21.739
21	1.169	72.91	24.50	17.86	34	22.907
22	1.179	73.53	26.00	19.12	36	24.310
23	1.188	74.10	27.25	20.19	37½	25.479
24	1.198	74.72	28.50	21.30	39½	26.647
25	1.208	75.34	30.00	22.60	41½	28.050

SPECIFIC GRAVITY OF SULPHURIC ACID—*Continued.*

Degrees Baumé.	Specific Gravity.	Weight of 1 Cubic Foot in Pounds	Per Cent O. V.	Pounds O. V. in 1 Cubic Foot.	Degrees Tw.	Per Cent H_2SO_4.
26	1.218	75.97	31.25	23.74	43½	29.219
27	1.229	76.65	32.75	25.10	46	30.621
28	1.239	77.28	34.00	26.28	48	31.790
29	1.250	77.96	35.50	27.68	50	33.192
30	1.261	78.65	37.00	29.10	52	34.595
31	1.272	79.33	38.37	30.44	54½	35.876
32	1.283	80.02	39.75	31.81	56½	37.166
33	1.295	80.77	41.25	33.32	59	38.569
34	1.306	81.46	42.62	34.72	61	39.850
35	1.318	82.20	44.00	36.17	63½	41.140
36	1.330	82.95	45.50	37.74	66	42.542
37	1.342	83.70	47.00	39.31	68½	43.945
38	1.355	84.51	48.50	40.99	71	45.347
39	1.368	85.32	50.00	42.66	73½	46.750
40	1.381	86.13	51.50	44.30	76	48.152
41	1.394	86.94	53.00	46.08	79	49.555
42	1.408	87.82	54.50	47.86	81½	50.957
43	1.421	88.63	56.00	49.63	84	52.360
44	1.436	89.56	57.50	51.50	87	53.762
45	1.450	90.44	59.00	53.36	90	55.165
46	1.465	91.37	60.50	55.28	93	56.567
47	1.479	92.25	62.00	57.50	96	57.970
48	1.495	93.24	63.50	59.21	99	59.372
49	1.510	94.18	65.00	61.22	102	60.775
50	**1.526**	**95.18**	**66.50**	**63.29**	**105**	**62.177**
51	1.542	96.17	68.00	65.40	108½	63.580
52	1.559	97.23	69.70	67.77	112	65.169
53	1.576	98.30	71.30	70.09	115	66.665
54	1.593	99.36	73.00	72.53	118½	68.255
55	1.611	100.48	74.60	74.96	122	69.751
56	1.629	101.60	76.20	77.42	126	71.247
57	1.648	102.79	77.87	80.04	129½	72.808
58	1.666	103.91	79.50	82.61	133	74.332
59	1.686	105.16	81.30	85.50	137	76.015
60	1.706	106.40	83.00	88.31	141	77.605
61	1.726	107.65	85.00	91.50	145	79.475
62	1.747	108.96	87.00	94.80	149½	81.345
63	1.768	110.27	89.00	98.14	153½	83.215
64	1.790	111.64	91.62	102.29	158	85.665
64½	1.801	112.33	93.12	104.60	160¼	87.067
64¾	1.807	112.70	94.00	105.94	161½	87.890
65	1.812	113.01	94.70	107.02	162½	88.544
65¼	1.818	113.39	95.66	108.47	163½	89.442
65½	1.824	113.76	96.80	110.12	164¼	90.508
65¾	1.830	114.14	98.33	112.23	166	91.939
66	1.835	114.45	100.00	114.45	167	93.500

STANDARDS ADOPTED.

Baumé Hydrometer
Modulus = 145.
66th degree = Sp. Gr. 1.835 at standard temperature, 60° F.
1 cu. ft. of water = 62.37 lbs. av.
H_2SO_4 = 100.

	H_2SO_4	O. V.	60
O. V.	93 50	100	120.48
60°	77.605	83	109
50°	62.18	66 5	80.12

APPROXIMATE BOILING-POINTS.

Acid.	F.	Acid.	F.
50°	291	63°	423
60°	378	64°	446
61°	388	65°	473
62°	403	66°	522

Per Cent 60°.	Pounds 60° in 1 Cubic Foot.	Per Cent 50°.	Pounds 50° in 1 Cubic Foot.
62 05	53.44	77 44	66.70
63 85	55.51	79 70	69.29
65 66	57.66	81 95	71 97
67 47	59.80	84 21	74 64
69 28	62 05	86 47	77.44
71 08	64 28	88 72	80.24
72.89	66 60	90 98	83.13
74.70	68 91	93 23	86 00
76.59	71 33	95 49	89 03
78.31	73 75	97 74	92 05
80.12	**76.26**	**100.00**	**95.18**
81 93	78.79	102 26	98 34
83.98	81.65	104 81	101.91
85.90	84 44	107.22	105.40
87.95	87 39	109 77	109.07
89 88	90 31	112 18	112 72
91 81	93 28	114 59	116 42
93.82	96 44	117 10	120.37
95 78	99 52	119 55	124.22
97 95	103 00	122.26	128 57
100 00	106 40	124 81	132 80
102 41	110 24	127 82	137.60
104 82	114 21	130 83	142.55
107.23	118 24	133 83	147.57
110 39.	123.24	137.77	153.81
114 10	128.94	142.41	160 94
120.48	137.89	150.38	172.11

ALLOWANCE FOR TEMPERATURE.

10° B.. ....46° F. = 1° B.
20° B............31.8° F. = 1° B.
30° B............30.25° F. = 1° B.
40° B............31.46° F. = 1° B.

50° B............34.69° F. = 1° B.
60° B............40.00° F. = 1° B.
66° B............43.24° F. = 1° B.

TABLE XLIII.—SPECIFIC GRAVITY OF HYDROCHLORIC ACID.

Specific Gravity at $\frac{15^\circ}{4^\circ}$ C. (in vacuo).	Degrees Baumé.	Degrees Twad.	100 Parts correspond to					
			HCl Per Cent.	18° Baumé Acid Per Cent.	19° Baumé Acid Per Cent.	20° Baumé Acid Per Cent.	21° Baumé Acid Per Cent.	22° Baumé Acid Per Cent.
1.000	0.0	0.0	0.16	0.57	0.53	0.49	0.47	0.45
1.005	0.7	1	1.15	4.08	3.84	3.58	3.42	3.25
1.010	1.4	2	2.14	7.60	7.14	6.66	6.36	6.04
1.015	2.1	3	3.12	11.08	10.41	9.71	9.27	8.81
1.020	2.7	4	4.13	14.67	13.79	12.86	12.27	11.67
1.025	3.4	5	5.15	18.30	17.19	16.04	15.30	14.55
1.030	4.1	6	6.15	21.85	20.53	19.16	18.27	17.38
1.035	4.7	7	7.15	25.40	23.87	22.27	21.25	20.20
1.040	5.4	8	8.16	28.99	27.24	25.42	24.25	23.06
1.045	6.0	9	9.16	32.55	30.58	28.53	27.22	25.88
1.050	6.7	10	10.17	36.14	33.95	31.68	30.22	28.74
1.055	7.4	11	11.18	39.73	37.33	34.82	33.22	31.59
1.060	8.0	12	12.19	43.32	40.70	37.97	36.23	34.44
1.065	8.7	13	13.19	46.87	44.04	41.09	39.20	37.27
1.070	9.4	14	14.17	50.35	47.31	44.14	42.11	40.04
1.075	10.0	15	15.16	53.87	50.62	47.22	45.05	42.84
1.080	10.6	16	16.15	57.39	53.92	50.31	47.99	45.63
1.085	11.2	17	17.13	60.87	57.19	53.36	50.90	48.40
1.090	11.9	18	18.11	64.35	60.47	56.41	53.82	51.17
1.095	12.4	19	19.06	67.73	63.64	59.37	56.64	53.86
1.100	13.0	20	20.01	71.11	66.81	62.33	59.46	56.54
1.105	13.6	21	20.97	74.52	70.01	65.32	62.32	59.26
1.110	14.2	22	21.92	77.89	73.19	68.28	65.14	61.94
1.115	14.9	23	22.86	81.23	76.32	71.21	67.93	64.60
1.120	15.4	24	23.82	84.64	79.53	74.20	70.79	67.31
1.125	16.0	25	24.78	88.06	82.74	77.19	73.74	70.02
1.130	16.5	26	25.75	91.50	85.97	80.21	76.52	72.76
1.135	17.1	27	26.70	94.88	89.15	83.18	79.34	75.45
1.140	17.7	28	27.66	98.29	92.35	86.17	82.20	78.16
1.142	18.0	..	28.14	100.00	93.95	87.66	83.62	79.51
1.145	18.3	29	28.61	101.67	95.52	89.13	85.02	80.84
1.150	18.8	30	29.57	105.08	98.73	92.11	87.87	83.55
1.152	19.0	..	29.95	106.43	100.00	93.30	89.01	84.63
1.155	19.3	31	30.55	108.58	102.00	95.17	90.79	86.32
1.160	19.8	32	31.52	112.01	105.24	98.19	93.67	89.07
1.163	20.0	..	32.10	114.07	107.17	100.00	95.39	90.70
1.165	20.3	33	32.49	115.46	108.48	101.21	96.55	91.81
1.170	20.9	34	33.46	118.91	111.71	104.24	99.43	94.55
1.171	21.0	..	33.65	119.58	112.35	104.82	100.00	95.09
1.175	21.4	35	34.42	122.32	114.92	107.22	102.28	97.26
1.180	22.0	36	35.39	125.76	118.16	110.24	105.17	100.00
1.185	22.5	37	36.31	129.03	121.23	113.11	107.90	102.60
1.190	23.0	38	37.23	132.30	124.30	115.98	110.63	105.20
1.195	23.5	39	38.16	135.61	127.41	118.87	113.40	107.83
1.200	24.0	40	39.11	138.98	130.58	121.84	116.22	110.51

TABLE XLIV.—NUMBER OF GALLONS IN CISTERNS AND TANKS.

Depth in Feet.	Diameter in Feet.								
	5	6	7	8	9	10	11	12	13
5	725	1060	1440	1875	2380	2,925	3,550	4,237	4,960
6	870	1270	1728	2250	2855	3,510	4,260	5,084	5,952
7	1015	1480	2016	2625	3330	4,095	4,970	5,931	6,944
8	1160	1690	2304	3000	3805	4,680	5,680	6,778	7,936
9	1305	1900	2592	3375	4280	5,265	6,390	7,625	8,928
10	1450	2110	2880	3750	4755	5,850	7,100	8,472	9,920
11	1595	2320	3168	4125	5230	6,435	7,810	9,319	10,913
12	1740	2530	3456	4500	5705	7,020	8,520	10,166	11,904
13	1885	2740	3744	4875	6180	7,605	9,230	11,013	12,896
14	2030	2950	4032	5250	6655	8,190	9,940	11,860	13,888
15	2175	3160	4320	5625	7130	8,775	10,650	12,707	14,880
16	2320	3370	4608	6000	7605	9,360	11,360	13,554	15,872
17	2465	3580	4896	6375	8080	9,945	12,070	14,401	16,864
18	2610	3790	5184	6750	8535	10,530	12,780	15,248	17,856
19	2755	4000	5472	7125	9010	11,115	13,490	16,095	18,848
20	2900	4210	5760	7500	9490	11,700	14,200	16,942	19,840

Depth in Feet.	Diameter in Feet.							
	14	15	16	18	20	22	24	25
5	5,765	6,698	7,520	9,516	11,750	14,215	16,918	18,358
6	6,918	8,038	9,024	11,419	14,100	17,059	20,302	22,030
7	8,071	9,378	10,528	13,322	16,450	19,902	23,680	25,701
8	9,224	10,718	12,032	15,225	18,800	22,745	27,070	29,372
9	10,377	12,058	13,536	17,128	21,150	25,588	30,454	33,043
10	11,530	13,398	15,040	19,031	23,500	28,431	33,838	36,714
11	12,683	14,738	16,544	20,934	25,850	31,274	37,222	40,385
12	13,836	16,078	18,048	22,837	28,200	34,117	40,606	44,056
13	14,989	17,418	19,552	24,740	30,550	36,960	43,990	47,727
14	16,142	18,758	21,056	26,643	32,900	47,374	47,374	51,398
15	17,295	20,098	22,260	28.546	35,250	42,646	50,758	55,069
16	18,448	21,438	24,064	30,459	37,600	25,489	54,142	58,740
17	19,601	22,778	25,568	32,352	39,950	48,332	57,520	62,411
18	20,754	24,118	27,072	34,255	42,300	51,175	60,910	66,082
19	21,907	25,458	78,576	36,158	44,650	54,018	64,294	69,753
20	23,060	26,798	30,080	38,062	47,000	56,861	67,678	73,424

For tanks that are tapering, measure the diameter four-tenths from the large end. The American standard gallon contains 231 cubic inches, or 8⅓ lbs. of pure water. A cubic foot contains 62.3 lbs. of water, or 7.48 gallons.

TABLE XLV.—VALUE OF COMMERCIAL PRODUCTS AS COMPARED WITH CHEMICALLY PURE PRODUCTS. BASIS, UNITY FOR THE C. P. SUBSTANCE.

Per Cent.	Value.	Per Cent.	Value.	Per Cent.	Value.	Per Cent.	Value.	Per Cent.	Value.
100	1.0000	80	1.2500	60	1.6667	40	2.5000	20	5.0000
99	1.0101	79	1.2658	59	1.6949	39	2.5641	19	5.2631
98	1.0204	78	1.2821	58	1.7241	38	2.6316	18	5.5555
97	1.0309	77	1.2987	57	1.7544	37	2.7027	17	5.8823
96	1.0417	76	1.3158	56	1.7857	36	2.7778	16	6.2500
95	1.0526	75	1.3333	55	1.8182	35	2.8571	15	6.6667
94	1.0638	74	1.3513	54	1.8519	34	2.9412	14	7.1429
93	1.0753	73	1.3699	53	1.8868	33	3.0303	13	7.6923
92	1.0869	72	1.3889	52	1.9231	32	3.1250	12	8.3333
91	1.0989	71	1.4085	51	1.9608	31	3.2258	11	9.0909
90	1.1111	70	1.4285	50	2.0000	30	3.3333	10	10.0000
89	1.1236	69	1.4493	49	2.0408	29	3.4483	9	11.1111
88	1.1364	68	1.4706	48	2.0833	28	3.5714	8	12.5000
87	1.1494	67	1.4925	47	2.1277	27	3.7037	7	14.2857
86	1.1628	66	1.5151	46	2.1739	26	3.8461	6	16.6666
85	1.1765	65	1.5385	45	2.2222	25	4.0000	5	20.0000
84	1.1905	64	1.5625	44	2.2727	24	4.1667	4	25.0000
83	1.2048	63	1.5873	43	2.3256	23	4.3478	3	33.3333
82	1.2195	62	1.6129	42	2.3809	22	4.5455	2	50.0000
81	1.2346	61	1.6393	41	2.4390	21	4.7619	1	100.0000

TABLE XLVI.—EQUIVALENT PRICES FOR SODA-ASH. (BASIS 48 PER CENT.)

Price per 100 Lbs. 48 Per Cent.	Price per 100 Lbs. 58 Per Cent.	Price per Ton (2,240 Lbs.). 48 Per Cent.	Price per Ton (2,240 Lbs.). 58 Per Cent.	Price per 1000 Kilos (2,204.6 Lbs.). 48 Per Cent.	Price per 1000 Kilos (2,204.6 Lbs.). 58 Per Cent.
$0.01	$0.0121	$0.224	$0.2706	$0.2205	$0.2664
.02	.0242	.448	.5413	.4409	.5328
.03	.0362	.672	.8120	.6614	.7992
.04	.0483	.896	1.0826	.8818	1.0656
.05	.0604	1.120	1.3533	1.1023	1.3319
.06	.0725	1.344	1.6240	1.3228	1.5983
.07	.0846	1.568	1.8946	1.5432	1.8647
.08	.0966	1.792	2.1653	1.7637	2.1311
.09	.1087	2.016	2.4360	1.9841	2.3975
.10	.1208	2.240	2.7066	2.2046	2.6639
.15	.1812	3.360	4.0600	3.3069	3.8958
.20	.2417	4.480	5.4133	4.4092	5.3278
.25	.3021	5.600	6.7666	5.5115	6.6597
.30	.3625	6.720	8.1200	6.6138	7.9917
.35	.4229	7.840	9.4733	7.7161	9.3236
.40	.4833	8.960	10.8266	8.8184	10.6556
.45	.5437	10.080	12.1800	9.9207	11.9875
.50	.6042	11.200	13.5333	11.0230	13.3194
.55	.6646	12.320	14.8866	12.1253	14.6514
.60	.7250	13.440	16.2400	13.2276	15.9833
.65	.7854	14.560	17.5933	14.3299	17.3153
.70	.8458	15.680	18.9466	15.4322	18.6472
.75	.9062	16.800	20.3000	16.5345	19.9792
.80	.9666	17.920	21.6533	17.6368	21.3111
.85	1.0271	19.040	23.0066	18.7391	22.6431
.90	1.0875	20.160	24.3600	19.8415	23.9750
.95	1.1479	21.280	25.7133	20.9137	25.3070
1.00	1.2083	22.400	27.0666	22.0460	26.6389
1.05	1.2687	23.520	28.4200	23.1483	27.9708
1.10	1.3292	24.640	29.7733	24.2506	29.3028
1.15	1.3896	25.760	31.1266	25.3529	30.6348
1.20	1.4500	26.880	32.4800	26.4552	31.9667
1.25	1.5104	28.000	33.8333	27.5575	33.2986
1.30	1.5708	29.120	35.1866	28.6598	34.6306
1.35	1.6312	30.240	36.5400	29.7621	35.9625

TABLE XLVIII.—EQUIVALENT PRICES FOR CAUSTIC SODA, PER LBS. (BASIS 60 PER CENT).

Price per 100 Lbs. 60 Per Cent.	Price per 100 Lbs. 70 Per Cent.	Price per 100 Lbs. 72 Per Cent.	Price per 100 Lbs. 74 Per Cent.	Price per 100 Lbs. 76 Per Cent.
$0.01	$0.0117	$0.012	$0.0123	$0.0127
.02	.0233	.024	.0246	.0253
.03	.0350	.036	.0370	.0380
.04	.0467	.048	.0493	.0507
.05	.0583	.060	.0617	.0630
.06	.0700	.072	.0740	.0760
.07	.0817	.084	.0863	.0887
.08	.0933	.096	.0987	.1130
.09	.1050	.108	.1100	.1140
10	.1167	.120	.1233	.1267
.15	.1750	.180	.1850	.1900
.20	.2333	.240	.2467	.2533
.25	.2916	.300	.3083	.3167
.30	.3500	.360	.3700	.3800
.40	.4667	.480	.4933	.5067
.50	.5833	.600	.6167	.6333
.60	.7000	.720	.7400	.7600
.70	.8167	.840	.8633	.8867
.80	.9333	.960	.9867	1.0133
.90	1.0500	1.080	1.1100	1.1400
1.00	1.1667	1.200	1.2333	1.2667
1.10	1.2833	1.320	1.3567	1.3933
1.20	1.4000	1.440	1.4800	1.5200
1.30	1.5167	1.560	1.6033	1.6467
1.40	1.6333	1.680	1.7267	1.7733
1.50	1.7500	1.800	1.8500	1.9000
1.60	1.8667	1.920	1.9733	2.0267
1.70	1.9833	2.040	2.0967	2.1533
1.80	2.1000	.160	2.2200	2.2800
1.90	2.2167	2.280	2.3433	2.4067
2.00	2.3333	2.400	2.4667	2.5333
2.10	2.4500	2.520	2.5900	2.6600
2.20	2.5667	2.640	2.7133	2.7867
2.30	2.6833	2.760	2.8367	2.9133
2.40	2.8000	2.880	2.9600	3.0400
2.50	2.9167	3.000	3.0833	3.1667
2.60	3.0333	3.120	3.2067	3.2933
2.70	3.1500	3.240	3.3300	3.4200
2.80	3.2667	3.360	3.4533	3.5467
2.90	3.3833	3.480	3.5767	3.6733
3.00	3.5000	3.600	3.7000	3.8000

TABLE XLVIII.—POUNDS OF TALLOW SAPONIFIED BY 100 POUNDS OF CAUSTIC LYE OF DIFFERENT DENSITIES, MADE OF THE GRADES OF CAUSTIC INDICATED. (LAMBORN.)

Degrees Bé.	77.5°	76°.	74°.	72°.	70°.	60°.	Degrees Twaddel.
10	46.78	45.86	41.66	43.45	42.24	36.20	15.0
11	52.22	5[illegible].21	49.85	48.51	47.17	40.42	16.6
12	57.14	56.03	54.55	53.08	51.59	44.24	18.2
13	62.00	60.80	59.20	57.59	56.01	47.99	20.0
14	67.30	66.00	64.25	62.52	60.78	52.10	21.6
15	71.85	70.46	68.60	66.74	64.88	55.62	[illegible]3.2
16	78.38	76.86	74.84	72.81	70.79	[illegible].67	25.0
17	84.59	82.94	80.76	78.57	76.40	65.48	26.8
18	90.02	88.26	85.94	83.62	81.28	69.90	28.4
19	96.79	94.91	92.31	89.91	87.42	74.92	30.4
20	102.64	100.6	97.95	95.30	92.64	79.39	32.4
21	108.1	106.0	103.1	100.4	97.61	83.66	34.2
22	113.61	11[illegible].4	1[illegible]8.5	105.5	102.6	87.92	36.0
23	119.8	117.4	113.4	111.3	108.1	92.72	38.0
24	126.2	123.8	120.5	117.2	113.9	97.70	40.0
25	132.71	130.1	126.7	123.3	119.9	102.7	42.0
26	139.8	137.2	133.6	129.9	126.3	108.2	44.0
27	147.1	144.2	139.8	136.7	132.8	115.9	46.2
28	153.00	150.1	146.1	142.1	138.1	118.4	48.2
29	161.7	158.6	154.4	150.3	146.1	125.2	50.4
30	169.07	165.8	161.4	157.0	152.6	130.8	52.6
31	177.2	173.8	169.2	161.3	159.7	136.9	54.8
32	184.3	180.7	176.0	171.2	166.4	142.7	57.0
33	191.6	187.9	183.0	178.0	173.1	148.4	59.4
34	198.6	194.7	189.5	184.5	179.8	153.7	61.6
35	205.9	201.9	196.6	191.3	186.0	159.3	64.0
36	213.8	209.6	204.1	198.6	193.2	165.5	66.4
37	223.0	218.7	212.9	207.2	201.5	172.7	69.0
38	231.9	227.4	221.4	215.1	209.4	179.5	71.4

Specific Gravity of Aqueous Solutions of Glycerin.—The most accurate numbers are those published by Lenz, Strohmer, Gerlach, Nicol, and Skalweit.[1] Lenz derived his table from a sample of chemically pure glycerin, the glycerin of which had been determined by ultimate analysis. Strohmer used crystallized glycerin rendered anhydrous by repeated pressing between folds of filter-paper. Gerlach used a sample of glycerin with boiling-point constant at 29° C., prepared by concentrating chemically pure glycerin of specific gravity 1.220. Morawski (J. S. C. I., 1889, 424) has controlled a few of the numbers given below by ultimate analysis. His results show that Lenz's figures are, as a rule, a little too high, those of Strohmer a little too low, while those of Gerlach and Skalweit agree among themselves and with chemical analysis. In the determination of the specific gravity of glycerin at temperatures less than normal, Hehner (J. S. C. I., 1889, 8) makes a correction of 0.00058 for each degree Centigrade.

TABLE XLIX.—SPECIFIC GRAVITY OF AQUEOUS SOLUTIONS OF GLYCERIN.

Glycerol. Per Cent.	Lenz.	Strohmer.	Gerlach.		Nicol.
	Specific Gravity at 12°–14° C. Water at 12° C.—1.	Specific Gravity at 17.5° C. Water at 17.5° C.—1.	Specific Gravity at 15° C. Water at 15° C.—1.	Specific Gravity at 20° C. Water at 20° C.—1	Specific Gravity at 20° C. Water at 20° C.—1.
100	1.2691	1.262	1.2653	1.2620	1.26348
99	1.2664	1.259	1.2628	1.2594	1.26091
98	1.2637	1.257	1.2602	1.2568	1.25832
97	1.2610	1.254	1.2577	1.2542	1.25572
96	1.2584	1.252	1.2552	1.2516	1.25312
95	1.2557	1.249	1.2526	1.2490	1.25052
94	1.2531	1.246	1.2501	1.2464	1.24790
93	1.2504	1.244	1.2476	1.2438	1.24526
92	1.2478	1.241	1.2451	1.2412	1.24259
91	1.2451	1.239	1.2425	1.2386	1.23990
90	1.2425	1.236	1.2400	1.2360	1.23720
89	1.2398	1.233	1.2373	1.2333	1.23449
88	1.2372	1.231	1.2346	1.2306	1.23178
87	1.2345	1.228	1.2319	1.2279	1.22907
86	1.2318	1.226	1.2292	1.2252	1.22636
85	1.2292	1.223	1.2265	1.2225	1.22365
84	1.2265	1.220	1.2238	1.2198	1.22094
83	1.2238	1.218	1.2211	1.2171	1.21823
82	1.2212	1.215	1.2184	1.2144	1.21552
81	1.2185	1.213	1.2157	1.2117	1.21281

SPECIFIC GRAVITY OF AQUEOUS SOLUTIONS OF GLYCERIN—*Cont'd.*

Glycerol. Per Cent.	Lenz.	Strohmer.	Gerlach.		Nicol.
	Specific Gravity at 12°–14° C. Water at 12° C. = 1.	Specific Gravity at 17.5 C. Water at 17.5° C. = 1.	Specific Gravity at 15° C. Water at 15° C. = 1.	Specific Gravity at 20° C. Water at 20° C. = 1.	Specific Gravity at 20° C. Water at 20° C. = 1.
80	1.2159	1.210	1.2130	1.2090	1.21010
79	1.2122	1.207	1.2102	1.2063	1.20739
78	1.2106	1.204	1.2074	1.2036	1.20468
77	1.2079	1.202	1.2046	1.2009	1.20197
76	1.2042	1.199	1.2018	1.1982	1.19925
75	1.2016	1.196	1.1990	1.1955	1.19653
74	1.1999	1.193	1.1962	1.1928	1.19381
73	1.1973	1.190	1.1934	1.1901	1.19109
72	1.1945	1.188	1.1906	1.1874	1.18837
71	1.1918	1.185	1.1878	1.1847	1.18565
70	1.1889	1.182	1.1850	1.1820	1.18293
69	1.1858	1.179	......	......	1.18020
68	1.1826	1.176	......	......	1.17747
67	1.1795	1.173	......	......	1.17474
66	1.1764	1.170	......	......	1.17201
65	1.1733	1.167	1.1711	1.1685	1.16928
64	1.1702	1.163	......	......	1.16654
63	1.1671	1.160	......	......	1.16380
62	1.1640	1.157	......	......	1.16107
61	1.1610	1.154	......	......	1.15834
60	1.1582	1.151	1.1570	1.1550	1.15561
59	1.1556	1.149	......	......	1.15288
58	1.1530	1.146	......	......	1.15015
57	1.1505	1.144	......	......	1.14742
56	1.1480	1.142	......	......	1.14469
55	1.1455	1.140	1.1430	1.1415	1.14196
54	1.1430	1.137	......	......	1.13923
53	1.1403	1.135	......	......	1.13650
52	1.1375	1.133	......	......	1.13377
51	1.1348	1.130	......	......	1.13104
50	1.1320	1.128	1.1290	1.1280	1.12831
45	1.1183	.....	1.1155	1.1145	1.11469
40	1.1045	.....	1.1020	1.1010	1.10118
35	1.0907	.....	1.0885	1.0875	1.08786
30	1.0771	.....	1.0750	1.0740	1.07469
25	1.0635	.....	1.0620	1.0610	1.06166
20	1.0498	.....	1.0490	1.0480	1.04884
15	1.0374	.....	......	......	1.03622
10	1.0245	.....	1.0245	1.0235	1.02391
5	1.0123	.....	......	......	1.01184
0	1.0000	.....	1.0000	1.0000	1.00000

TABLE L.—SPECIFIC GRAVITY OF AQUEOUS SOLUTIONS OF GLYCERIN.

(SKALWEIT.)

Glycerol. Per Cent.	Specific Gravity at 15° C.	Glycerol. Per Cent.	Specific Gravity at 15° C.	Glycerol Per Cent.	Specific Gravity at 15° C.	Glycerol. Per Cent.	Specific Gravity at 15° C.
0	1.0000	25	1.0620	50	1.1290	75	1.1990
1	1.0024	26	1.0646	51	1.1318	76	1.2017
2	1.0048	27	1.0672	52	1.1346	77	1.2044
3	1.0072	28	1.0698	53	1.1374	78	1.2071
4	1.0096	29	1.0724	54	1.1402	79	1.2098
5	1.0120	30	1.0750	55	1.1430	80	1.2125
6	1.0144	31	1.0777	56	1.1458	81	1.2152
7	1.0168	32	1.0804	57	1.1486	82	1.2179
8	1.0192	33	1.0831	58	1.1514	83	1.2206
9	1.0216	34	1.0858	59	1.1542	84	1.2233
10	1.0240	35	1.0885	60	1.1570	85	1.2260
11	1.0265	36	1.0912	61	1.1599	86	1.2287
12	1.0290	37	1.0939	62	1.1628	87	1.2314
13	1.0315	38	1.0966	63	1.1657	88	1.2341
14	1.0340	39	1.0993	64	1.1686	89	1.2368
15	1.0365	40	1.1020	65	1.1715	90	1.2395
16	1.0390	41	1.1047	66	1.1743	91	1.2421
17	1.0415	42	1.1074	67	1.1771	92	1.2447
18	1.0440	43	1.1101	68	1.1799	93	1.2473
19	1.0465	44	1.1128	69	1.1827	94	1.2499
20	1.0490	45	1.1155	70	1.1855	95	1.2525
21	1.0516	46	1.1182	71	1.1882	96	1.2550
22	1.0542	47	1.1209	72	1.1909	97	1.2575
23	1.0568	48	1.1236	73	1.1936	98	1.2600
24	1.0594	49	1.1263	74	1.1963	99	1.2625
						100	1.2650

TABLE LI.—SATURATED STEAM.

Table showing steam-pressure in pounds per steam-gauge; the absolute pressure per square inch measured from a vacuum; inches of mercury and temperature in degrees Fahrenheit.

Pressure per Steam-gauge.	Total Pounds.	Inches of Mercury.	Temperature in Degrees Fahrenheit.	Pressure per Steam-gauge.	Total Pounds.	Inches of Mercury.	Temperature in Degrees Fahrenheit.
....	1	2.03	102.1	45.3	60	122.16	292.7
....	2	4.07	126.3	55.3	70	142.52	302.9
....	3	6.01	141.6	65.3	80	162.88	312.0
....	4	8.14	153.1	75.3	90	185.24	320.2
....	5	10.18	162.3	85.3	100	203.60	327.9
....	6	12.21	170.2	95.3	110	223.96	334.6
....	7	14.25	176.9	105.3	120	244.32	341.1
....	8	16.28	182.9	115.3	130	264.68	347.2
....	9	18.32	188.3	125.3	140	285.04	352.9
....	10	20.36	193.3	135.3	150	305.40	358.3
....	11	22.39	197.8	145.3	160	325.75	363.4
....	12	24.43	202.0	155.3	170	346.11	368.2
....	13	26.46	205.9	165.3	180	366.47	372.9
0	14.7	28.50	212.0	175.3	190	386.83	377.5
.3	15	30.54	213.1	185.3	200	407.19	381.7
5.3	20	40.72	228.0	205.3	220	447.90	389.9
10.3	25	50.90	240.1	225.3	240	488.62	397.5
15.3	30	61.08	250.4	245.3	260	529.34	404.5
25.3	40	81.43	267.3	265.3	280	570.06	411.2
35.3	50	101.79	281.0	285.3	300	610.78	417.5

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